CHAPTER I
Introduction

Cotton is still the "king" of fibers because most of the world's apparel is made of cotton. It has fairly good strength, and it is considered to provide comfortable textile fabrics due to the fiber's good moisture absorption and wicking properties. Cotton fabrics, however, have a tendency to wrinkle badly and have poor smooth drying properties after laundering. Under distortion and moist conditions, the hydrogen bonds that hold the cellulose chains together are ruptured, and then the chains slide to minimize the stress within the fibers. This phenomenon causes the hydrogen bonds to reform in a new position after removal of the distorting force. The rupture and reformation of hydrogen bonds cause wrinkle problems on cotton or cotton-blend fabrics (Andrews, 1992, 1995; Cooke & Weighmann, 1982 a&b; Smith & Block, 1982 a&b). To improve those performance properties, cotton or cotton-blend fabrics often are given a chemical treatment called durable press finishing. This treatment involves the use of crosslinking agents, which can covalently crosslink with the adjacent cellulose chains within cotton fibers. The new crosslinking bonds formed in the durable press chemical finishing process are stronger than the former hydrogen bonds. The new crosslinks hold when the fabric is under distortion and moist conditions; the bonds pull the cellulose chains back into position after removal of a distorting force so that the fabric resists wrinkling.

Durable press (DP) chemical finishes applied to cotton fabrics in the presence of appropriate catalysts impart wrinkle resistance, shrinkage resistance, and smoothness after drying. Cotton fabrics treated with DP finishes will retain their dimensions, smooth appearance, and shape while in use and after machine washing and drying. The common crosslinking agents in present usage impart not only crease resistance and smooth drying properties, but also deleterious effects on other properties such as strength, color fastness, and softness.

The release of formaldehyde vapors is another problem with those agents. The most widely used crosslinking agents in DP finishes have been N-methylol agents or N-methylolamides because of their efficiency and low price. These reactants fall in the category of formaldehyde reactants (Cooke & Weigmann, 1982a; Peterson, 1987). The achievement of good DP properties and of the least formaldehyde release from cotton fabrics treated with formaldehyde reactants depends on the reactant types, the catalyst types, the time and temperature of cure, the condition of the treated fabrics, and the additives in the impregnating bath (Cooke & Weigmann, 1982 a&b). To achieve the best DP finishes and the least formaldehyde release of N-methylol compounds, all these factors must be controlled carefully.

Peterson (1983, 1987) classified formaldehyde reactants into three types according to reactivity and rates of hydrolysis. In group 1, urea, dihydroxymethylethyleneurea, and one of the hydroxymethylated propyleneureas have high reactivity and poor hydrolysis stability. In group 2, uron, carbamate, and dihydroxymethyl-4-methoxy-5,5-dimethylpropyleneurea have medium

reactivity and hydrolysis stability. In group 3, dihydroxymethyl-4,5-dihydroxyethyleneurea (DMDHEU) and glyoxal diureine have low reactivity and good resistance to hydrolysis. Reactants in group 3 provide durable press fabrics with low formaldehyde release.

Acids catalyze the reaction of N-methylol compounds with cellulose. The stronger the acid, the faster the reaction. Selecting the proper catalysts is important because stronger acids are required to catalyze the reaction of some less reactive crosslinking reagents, but the stronger the acid, the more damage to the cellulosic fibers through hydrolysis. The most commonly used catalysts for N-methylol compounds are zinc nitrate, magnesium chloride, or a combination of magnesium chloride and citric acid. Cooke and Weigmann (1982b) mentioned that zinc nitrate is a more effective catalyst than magnesium chloride, but it causes yellowing of white fabric and changes the shades of some fabric colors. A combination of magnesium chloride and citric acid is sometimes used instead of zinc nitrate. Thus, catalysts' effects on discoloration and strength deterioration of the treated fabrics are among the criteria for selection of a suitable catalyst.

Temperature and time of cure are important variables to control so as to obtain good crosslinking and low formaldehyde release in treated cotton fabrics. Fabrics treated with urea-formaldehyde (UF) and dihydroxymethylethyleneurea (DMEU) are easy to cure; however, cotton fabrics treated with these reactants need to be cured as soon as the fabric is dried, so that the reactants will not self-crosslink. DMDHEU and carbamate are in the hard-to-cure category where fabric curing temperature must exceed 130 °C (Cooke & Weigmann, 1982b); therefore, fabrics can be left for a long time before post curing. This feature is easier to handle in textile finishing. Residual alkali on fabrics being treated is an important aspect of the fabric condition. Alkali could hinder the curing of the formaldehyde reactants, and thus could increase the formaldehyde release (Cooke & Weigmann, 1982b). As well, any additives in the finishing formulation, such as softeners, hand builders, soil repellents, and formaldehyde scavengers, should be carefully controlled and tested to determine the effect on crosslinking and on formaldehyde release (Cooke & Weigmann, 1982b).

The formaldehyde reactants in greatest use as the crosslinking agents for DP finishes, though efficient and inexpensive, have two serious disadvantages (Choi, 1992). First, they release formaldehyde vapors during DP finishing, storage, and consumer use. The Occupational Safety and Health Administration (OSHA) lists formaldehyde as a hazardous and toxic substance (Andrews, 1995), and has set the upper limit for formaldehyde in air at 0.75 parts per million average over an eight-hour work shift. Several studies by the Chemical Industry Institute of Toxicology show that formaldehyde is a carcinogen to animals. To humans, it is a severe eye irritant, a mucous membrane irritant, and a skin irritant, and it is toxic if ingested (Cooke & Weigmann, 1982a). Secondly, formaldehyde treated fabrics suffer a major loss of such mechanical properties as tensile strength, tear strength, and abrasion resistance due to two key contributing factors. One factor is the fiber degradation caused by the acid catalysts at elevated temperatures. The other is the restriction of stress distribution within the fibers due to the crosslinked sites. The loss of mechanical

properties also occurs with some nonformaldehyde reactants, such as polycarboxylic acids.

Largely because of concern about formaldehyde hazards to workers in the textile industry and also to consumers, formaldehyde-free crosslinking agents for producing durable press properties are of interest to replace DMDHEU, the conventional finishing agent for DP finishes. Researchers at the Southern Regional Research Center (SRRC), New Orleans, LA, have been vigorously developing new and modified resins, with the proper catalysts, that can serve as formaldehyde-free durable press agents (Welch, 1988). Polycarboxylic acids (PCA), which are nonformaldehyde reactants, are possible replacements for the conventional finishing reactant. The main advantages of PCA are that they are formaldehyde-free, do not have a bad odor, and produce a very soft fabric hand. Loss of mechanical properties is a problem, however.

Research at SRRC has shown that the most important PCA reactants for the textile industry today are butanetetracarboxylic acid (BTCA) and citric acid (CA). The best results in DP finishes with PCA have been obtained with BTCA in the presence of sodium hypophosphite as the catalyst (Welch, 1988; Welch & Andrews, 1989 a&b). BTCA, in the presence of sodium hypophosphite, provides the same level of durable press performance and finish durability in laundering as does the conventional DMDHEU reactant. Yet, the high cost of BTCA is an obstacle to mills' decisions to use BTCA as replacement for conventional DP reactants.

Citric acid (CA) is another candidate to replace DMDHEU. The advantages of CA over other PCA are low cost, proven lack of toxicity, and ready availability (Andrews, 1989, 1990). However, CA in the presence of sodium hypophosphite as a catalyst does not provide the same level of DP properties as does DMDHEU reactant. One, two, three-propanetricarboxylic acid (TCA) provides good DP properties, but it needs a high percentage on weight of bath (% o.w.b.) to achieve good DP performance, and its cost is high (Andrews, 1990; Andrews & Trask-Morrell, 1991). PCA reactants can solve the problem of formaldehyde release from finished cotton fabrics. Further research is needed to bring about reduction in the cost of DP finishing with BTCA and TCA and in the loss of mechanical properties of cotton fabrics treated with PCA reactants.

Durable press finishing agents can be applied on cotton fabrics by a technique called the pad-dry-cure method. Pad-dry-cure is the conventional technique for applying durable press finishing agents to cotton fabrics because of its simplicity and ease. The fabric is immersed, for 5-10 minutes, in the aqueous solution containing a DP finish, curing catalyst, fabric softener, wetting agent, and water. Then, the fabric is padded through squeeze rolls to give a specified wet pick-up, reported as percent on weight of fabric. After that, the fabric is dried and cured for a specified time at a specified temperature. The pad-dry-cure technique results in fabric with good DP properties, but with high losses in tensile and tear strength and in abrasion resistance.

Some researchers (Bertoniere, Blouin, Martin & Rowland, 1974; Choi, 1992; Hollies & Getchell, 1967; Rowland, Bertoniere & Martin, 1974; Shin, Hollies & Yeh, 1989) have proposed DP finishing by the polymerization-

crosslinking (PC) treatment with a wet fixation process. PC treatment with wet fixation uses a combination of two resin components. The first component, called a polymer builder, polymerizes itself under the treatment conditions and keeps the fibers in swollen state. The second component, known as a crosslinker, contributes moderately to fiber swelling when wet fixed with a polymer builder, and it provides the crosslink formation with cellulose. The main way that the wet fixation process differs from conventional pad-dry-cure is the step in which the resin impregnated fabric is held in the moist state for a certain time at a certain temperature. The PC treatment used with a wet fixation process could improve breaking strength and abrasion resistance at a high level of DP properties because it allows time for resin penetration and deposition inside the fibers. A main disadvantage of the wet fixation process is timing. Any fabric treated with this process has to be wrapped in a plastic bag for many hours at room temperature to allow a reaction to occur effectively. PC treatment with the pad-dry-cure process can be done by adding an initiator to accelerate a reaction to occur faster than that with the wet fixation process. The duration time of PC treatment with pad-dry-cure is much shorter than that in PC treatment with wet fixation, so the effect of oxygen scavenger on the polymerization reaction may be less severe with pad-dry-cure. PC treatment normally employs formaldehyde based monomers such as melamine and N-methylolacrylamide, which release formaldehyde. Little has been done to examine alternative PC treatments that do not generate formaldehyde vapors.

PC treatment may be a method for achieving a balance of durable press and mechanical properties when used with PCA reactant combinations. Good performance properties in cotton fabrics may result from PC treatment used with pad-dry-cure of unsaturated carboxylic acids, such as acrylic acid (AA), maleic acid (MA), itaconic acid (IA), or a mixture of any two of these acids, as polymerizing resins or polymer builders, and BTCA, TCA, or CA, as crosslinkers. The mixture of two resin components of polycarboxylic acid reactants in the PC treatment with pad-dry-cure, to improve durable press properties of finished cotton fabrics, is a unique and new research area in wrinkle resistance.

The purpose of this research is to examine the mechanical and DP properties of cotton fabric finished by a polymerization-crosslinking process with pad-dry-cure, using BTCA/IA/AA combinations; the mixture of IA/AA is used as a polymer builder, and BTCA is used as a crosslinker. BTCA/IA/AA combinations were selected as the focus of the research, because, in a preliminary analysis conducted in this research, the BTCA/IA/AA combinations provided better results in wrinkle recovery angles of the finished fabric than did the other fabric finishing chemicals that were analyzed. Pad-dry-cure is used because it is the most common application technique for durable press finishing. In addition, the timing to finish the fabrics with the pad-dry-cure process is faster than that of the wet fixation processes. The results in this research were analyzed to assess the optimal treatment conditions for obtaining a balance of the mechanical and DP properties. The overall goals of this research were (a) to investigate a new BTCA/IA/AA combination of PCA for DP finishing which could provide good DP properties, compared with the DP properties resulting from the conventional

DMDHEU reactant or BTCA reactant alone, and which also could avert the major loss of mechanical properties of the treated cotton fabric, and (b) to find a cost-efficient process for using BTCA in DP finishing.

CHAPTER II
Literature Review

Fabric finishing consists of mechanical, chemical, and/or thermal processes, which are applied to fabrics to produce or impart specific performance properties that the fabrics lack altogether or lack at the desired performance level. Durable press finishing is one of the many types of fabric finishing. Apparel fabrics made from cellulose, regenerated cellulose, and blends of those with synthetic fibers (most commonly polyester) have a tendency to wrinkle badly after washing and tumble drying and also during wearing. Durable press finishing is a chemical process used to improve such fabrics' wrinkle resistance, or so-called durable press performance.

This chapter reviews the chemical structure of cellulose and the morphology of cotton fiber, literature concerning durable press finishes, application techniques, evaluation of fabric performance as to durable press properties, and also research pertinent to all those topics.

Cellulosic Fibers

Cellulosic fibers include cotton, linen, jute, ramie, hemp, and others, but cotton has become the most important textile fiber because it has fairly good strength, softness, moisture absorbency, and pliability. The structure of cotton fiber can be viewed in two directions: longitudinal and cross-sectional. The longitudinal view reveals that the fiber has a ribbon-like structure with twists, called convolutions, at irregular intervals along the fiber. Cross-sectionally, the fiber is kidney-shaped with a central hollow core known as the lumen (Cook, 1984; Joseph, 1988; Smith & Block, 1982a). Cotton fibers are composed of an outer cuticle, a primary wall, a secondary wall, and a central core known as the lumen. The cuticle surrounds the primary wall and is a thin hard shell, which protects the fibers from bruising during growth. The primary wall is tough and contains wax, protein, and pectinaceous substances as well as cellulose. It acts as a protective layer, forming the shell of the fiber during its early days of growth inside the boll. The secondary wall is almost pure cellulose and represents about 90 per cent of the total fiber weight. Cellulose in the cotton fiber's secondary wall is arranged in fibrils that are packed alongside each other. The secondary wall provides flexibility. The lumen provides liquid nutrients and protoplasm while the plant is growing.

Cotton fiber is almost 100 per cent cellulose. Cellulose is a high molecular weight polymer and is a substance of polysaccharide consisting of 2,000 or more glucose units in the molecule. The cellulose polymer forms by the condensation of glucose molecules, in which cellobiose is the repeat unit (Smith & Block, 1982a). The cellobiose repeat unit is composed of two beta-glucose monomer units linked by an oxygen atom that is in the same plane as the glucose rings. This structure allows the molecular chains of cellulose to pack together in parallel rows within the fibrils of the fiber. This alignment or orientation of the cellulose molecules in a regularized pattern forms the crystalline region of the cotton fiber, while the random arrangement of cellulose

molecules forms the amorphous region. The relative amounts of crystalline and amorphous cellulose influence the properties of cellulosic fiber. The crystallinity of cotton fiber is between 85 and 95 per cent, giving the cotton fiber moderate strength and good abrasion resistance.

The glucose ring in cellulose, containing one oxygen and five carbon atoms, has three hydroxyl groups in its structure. Because of the hydroxyl groups, cotton has the ability to absorb about one-fourth of its weight in water. This means that a cotton fabric will pick up body moisture and transport the moisture along the yarns to the outer surface of the fabric. The moisture transfer ability contributes to the good thermal comfort properties of cotton fabrics. A disadvantage of the hydrophilic nature of cotton is the tendency to easily pick up water-borne stains, such as coffee or juices, and trap the colorants of the stains in the fiber after the water evaporates. The major problem of the hydrophilic nature of cotton fiber is the tendency for fabrics to wrinkle badly. Therefore, cotton fabrics require durable press finishing to provide wrinkle resistance at the level consumers seek.

Durable Press Finishes

Durable press finishes are often classified according to the types of formaldehyde or nonformaldehyde chemicals used as the crosslinking agents (Cooke & Weigmann, 1982a; Peterson, 1987).

Formaldehyde Durable Press Finishes

Researchers in England at the Tootal Broadhurst Lee Co. Ltd., in 1928, were the first to investigate the development of a no-iron cellulosic fabric (Marsh, 1962; Ryan, 1971). The chemists discovered that molecules of phenol-formaldehyde resin could react together to form larger molecules; they found the same with urea-formaldehyde resin. A solution of those two resins, with a suitable catalyst, was applied to cotton fabric and then the fabric was dried and cured. Upon so treating the fabric, the molecules that had resulted from combining the two formaldehyde compounds could react inside the cellulosic fibers and form macromolecules within the fibers. These macromolecules resulted in the cotton fabric becoming more wrinkle resistant.

Phenol-formaldehyde was the first resin to be used in commercial durable press finishes (Marsh, 1962). The reaction of phenol and formaldehyde produces three reactive positions. These reactive groups permit the formation of a lattice structure under appropriate chemical reaction conditions. Phenol-formaldehyde could serve as an effective durable press finish if it did not cause severe discoloration of white cotton fabric and did not produce severe formaldehyde vapors.

Aminoplasts were studied during the same time as phenol-formaldehyde in 1928. The aminoplasts consisted of two major types of formaldehyde condensates: urea-formaldehyde and melamine-formaldehyde. These aminoplasts are able to form three-dimensional polymers by self-crosslinking, as does phenol-formaldehyde. Aminoplasts have the advantage of not discoloring treated cotton fabrics (Marsh, 1962). On the other hand, urea-formaldehyde reacts with hypochlorite bleach, which is a common laundry additive; the reaction

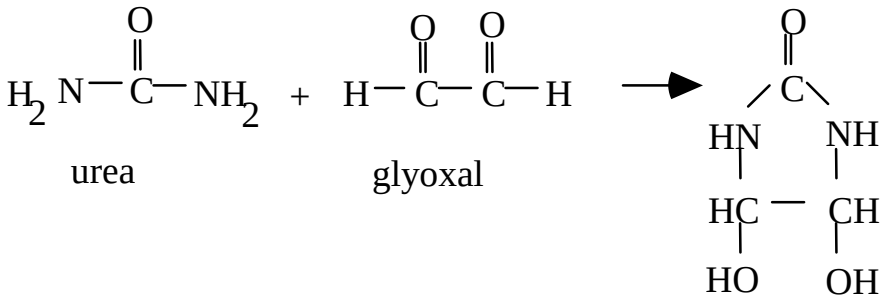
produces chloramines, which decompose in water under heat to liberate hydrochloric acid. Hydrochloric acid causes severe tendering of cotton fabrics. The chloramines produced by the reaction of chlorine with free -NH groups remaining in the resin structure of a melamine-formaldehyde system are more stable than those produced from a urea-formaldehyde system. With greater stability, the decomposition to liberate hydrochloric acid and the associated fabric tendering are less likely. Fabric yellowing during bleaching does occur, however, with the use of melamine-formaldehyde. Therefore, cellulosic fabrics treated with aminoplasts must be bleached only with nonchlorine agents, such as hydrogen peroxide, to avoid fabric tendering and yellowing (Ryan, 1971).

Phenol-formaldehyde and aminoplasts each form three-dimensional polymers by self-condensation before they crosslink with hydroxyl groups in cellulosic fibers. Such chemicals that self-condense before crosslinking are called resins. Another type of chemical used in durable press finishing tends to react directly with the cellulosic fibers, rather than self-condensing beforehand. This type of chemical is called a reactant. The commercially important reactants used in durable press finishes are dihydroxymethylethyleneurea (DMEU), carbamates, and dihydroxymethyl-4, 5-dihydroxyethyleneurea (DMDHEU).

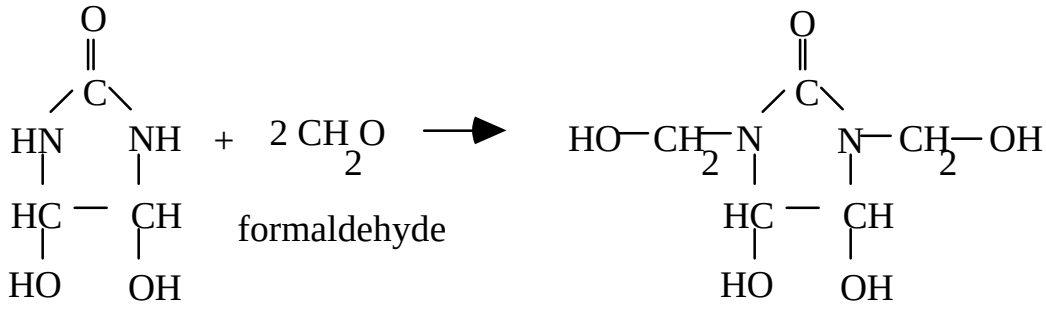
DMEU is made by reacting urea and ethylenediamine to form ethyleneurea, and then ethyleneurea containing two -NH groups is reacted with two moles of formaldehyde to give the DMEU. DMEU was widely used in the 1950s as a wash and wear finish. It is easily cured at around 90 to 100 °C. It provides good wrinkle recovery, but its application has diminished greatly in recent years because it has poor resistance to chlorine and hydrolysis (Cooke & Weigmann, 1982a; Marsh, 1962).

The starting compound for producing carbamates generally contains some alkyl group such as methyl, ethyl, hydroxyethyl, methoxyethyl, isopropyl, or isobutyl. The methyl and ethyl carbamates are carcinogenic and no longer used in durable press finishes. Carbamates react with formaldehyde to form N-methylol derivatives. This reaction is difficult to drive to completion, so it tends to cause high formaldehyde release. The carbamates give finished fabric with good durable press properties, and they do not cause discoloration of white fabrics. In addition, they have good resistance to chlorine and to hydrolysis, but they are highly prone to release formaldehyde vapors.

DMDHEU is the most important formaldehyde reactant used at present for durable press finishing. DMDHEU is made by reacting urea and glyoxal to form 4,5-dihydroxyethylene urea, which is then reacted with two moles of formaldehyde to yield DMDHEU. The formation of DMDHEU is shown below.



urea glyoxal 4,5-dihydroxyethyleneurea



formaldehyde DMDHEU

DMDHEU has relatively low free formaldehyde release, which makes it easy to handle in a finishing plant. It has good hydrolysis resistance and chlorine resistance, so that the cellulose crosslinks are more resistant to home laundering conditions than occurs with DMEU. Moreover, DMDHEU does not affect the light fastness of reactive and direct dyed materials, which are very common among cellulosic fabrics (Cooke & Weigmann, 1982a; Marsh, 1962).

The most widely used crosslinking agents in durable press finishes are the N-methylol compounds because of low expense, easy application, and excellent to good durable press properties; however, a major problem of these finishing agents is formaldehyde release. N-methylol compounds release formaldehyde vapors during durable press finishing, storage, and consumer use. Deichmann and Gerarde (1969) reported that inhalation of formaldehyde vapors may result in severe irritation and edema of the upper respiratory tract, burning and stinging of the eyes, and headache. Direct skin contact with formaldehyde causes irritation, dermatitis, discoloration and necrosis. The severity of these symptoms depends on the degree of exposure. Ingestion of formaldehyde induces severe gastroenteric changes and distress. Several studies by the Chemical Industry Institute of Toxicology showed that formaldehyde is a carcinogen to animals. To humans, it is a severe eye irritant, a mucous membrane irritant, and a skin irritant, and it is toxic if ingested (Cooke & Weigmann, 1982a). The Occupational Safety and Health Administration (OSHA) lists formaldehyde as a hazardous and toxic substance. The OSHA has lowered its permissible exposure limit for formaldehyde in air from 1.0 to 0.75 parts per million for an eight hour period (Andrews, 1995). Because of concern about

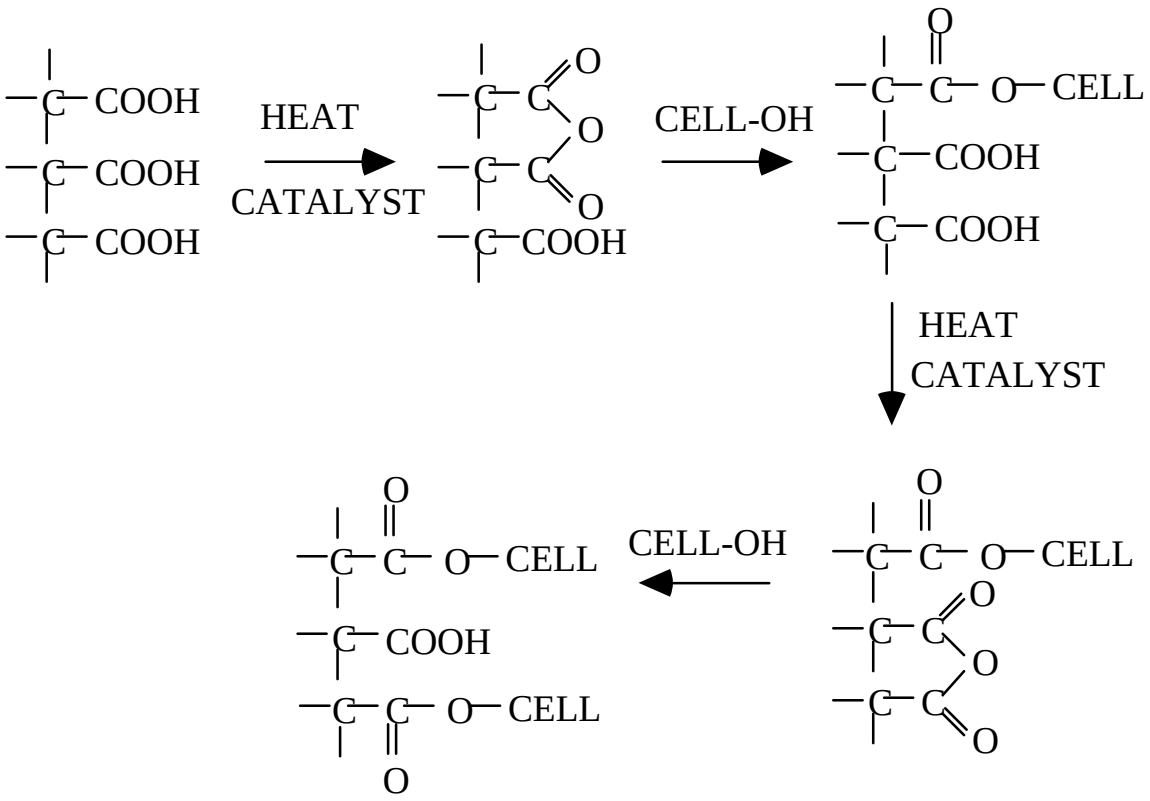
hazard from formaldehyde to workers in the textile industry and also to consumers, nonformaldehyde reactants for producing durable press properties are of interest to investigate for replacing the conventional N-methylol reactants, especially DMDHEU.

Nonformaldehyde Durable Press Finishes

N,N'-dimethyldihydroxyethyleneurea (DMeDHEU) and 1,3-dimethyl-4,5-dihydroxyethyleneurea (DHDMI) are two examples of nonformaldehyde reactants. The 4,5-ring hydroxyls of both reactants can react with cellulose hydroxyls and form the ether linkage, as do the N-methylol compounds. Major problems of these reactants are that they are hard-to-cure and more expensive than DMDHEU, have poor durable press properties, and cause yellowing on treated white fabrics (Cooke & Weigmann, 1982a). In recent years, the polycarboxylic acids class of nonformaldehyde agents has been developed for new formaldehyde-free durable press finishes for cotton fabrics to replace the conventional N-methylol compounds, especially DMDHEU. The development of polycarboxylic acids used as durable press finishes is reviewed below.

Polycarboxylic acids. Gagliardi and Shippee (1963) were the first to study the crosslinking of cellulose with polycarboxylic acids. They found that citric acid, tetracarboxylbutane, and diglycolic acid under a long curing time at high temperature increased fabric wrinkle resistance; however, low yield of crosslinking esterification within cellulosic fibers and high strength loss resulted from the high temperature and long curing time. The use of a mixture of phosphoric acid and urea as a catalyst system was not effective to achieve sufficient ester crosslinking. The researchers suggested that catalyst systems specific to the polycarboxylic acids may produce effective crosslinking at moderate temperatures and curing times.

Researchers at the Southern Regional Research Center (SRRC) in New Orleans have been developing new polycarboxylic acids, with the proper catalysts, that can serve as nonformaldehyde durable press finishes for use on cotton fabrics (Andrews, Welch & Trask-Morrell, 1989; Welch, 1990; Welch & Andrews, 1989 a&b, 1990). Polycarboxylic acids react with hydroxyl groups of cellulose to form ester crosslinks connecting adjacent cellulose chains in a three-dimensional network inside the cellulosic fibers. The researchers have reported a high degree of wrinkle resistance as well as smooth drying properties in cotton fabrics treated with polycarboxylic acids having three or more carboxyl groups, in the presence of alkali metal salts of a phosphorus-containing acid such as sodium hypophosphite, disodium phosphite, or monosodium phosphate. According to those scientists, polycarboxylic acids, in the presence of alkali metal salts of inorganic phosphorous-containing acids, react with cellulose via an anhydride mechanism. In the presence of heat and catalyst, two carboxylic groups form a five-membered anhydride ring and release one molecule of water. The cyclic anhydride reacts with cellulose to form an ester and regenerate one carboxylic acid group. The following is the mechanism of the formation of ester crosslinks in cotton treated with a polycarboxylic acid, as presented by scientists at SRRC (Andrews & Trask-Morrell, 1991; Welch, 1994).



As illustrated, each polycarboxylic acid molecule must have at least three carboxylic acid groups in order to provide effective ester crosslinking with cellulose found in textile fibers. Such effective crosslinking agents include 1,2,3,4-butanetetracarboxylic acid, 1,2,3-propanetricarboxylic acid, and citric acid, each having at least three carboxylic groups.

Researchers (Yang, 1991; Yang & Wang, 1996) have used Fourier transform infrared (FT-IR) spectroscopy to study the esterification mechanism of polycarboxylic acids with cotton cellulose. FT-IR spectroscopy detects anhydride carbonyl band absorbance and ester carbonyl absorbance in cotton treated with a polycarboxylic acid. The anhydride intermediate is chemically reactive. It can convert to carboxylic acid under moist conditions. In order to detect the anhydride intermediate, the treated sample must be analyzed immediately after the treatment so as to prevent the conversion of the anhydride to carboxylic acid due to moisture in the atmosphere. FT-IR spectroscopic data supported that cellulose esterification by a polycarboxylic acid occurs in two steps. The first step is the formation of a five-membered cyclic anhydride ring by the dehydration of two carboxyl groups. The second is the reaction between cellulose and the anhydride intermediate to form an ester linkage. FT-IR spectroscopic data reported by Yang and Wang (1996) show that BTCA has the highest ester carboxyl band absorbance among the seventeen polycarboxylic acids studied. The high ester carbonyl band absorbance indicated the ability of BTCA to form anhydride immediately and then, at its first carboxyl group, to esterify cellulose. The ester carbonyl band absorbance for BTCA-treated fabric gradually increases from 150 °C to 210 °C, which indicates that the number of carboxyl groups esterifying cellulose increases when the curing temperature increases. The

second carboxyl group of BTCA is able to esterify cellulose as effectively as its first carboxyl group, so the anhydride intermediates can further esterify cellulose, which makes BTCA a highly effective nonformaldehyde crosslinking agent for cotton cellulose.

The pH affects the formation of ester crosslinks in cotton treated with a polycarboxylic acid. Yang (1992; 1993) applied FT-IR spectroscopy in studying the effect of pH on BTCA used in durable press finishing of cotton fabrics. He found that the amount of the cyclic anhydride intermediate formed in cotton treated with BTCA decreases as the pH of the finish bath increases from 1.5 to 4.5; therefore, the cyclic anhydride intermediate formation is accelerated by increased proton concentration of the finish bath. Conversely, cellulose esterification increases as the pH of the bath increases from 1.5 to 3.5, although it then decreases when pH increases above 3.5. Due to the counter effect of pH on the cyclic anhydride intermediate formation and the cellulose esterification, it is necessary to choose the optimum pH range of a finish bath to achieve the most effective crosslinking by a polycarboxylic acid. Yang (1992) suggested an optimum pH range of 2.04 to 2.79 for the most effective esterification of cotton cellulose treated with BTCA.

BTCA is the most effective polycarboxylic acid found to date in terms of durable press finish performance and durability, as long as it is applied with one of a number of alkali metal salts of phosphorous-containing acids, such as sodium hypophosphite, disodium phosphite, or monosodium phosphate (Welch & Andrews, 1989 a&b; Welch, 1990, 1994). Sodium hypophosphite is the most effective curing catalyst for durable press finishing with BTCA, but it has the disadvantages of high cost and a tendency to produce shade changes in sulfur dyes. Disodium phosphite is less expensive and has no tendency to produce shade changes in dyed fabrics, so disodium phosphite is a recommended catalyst for use with BTCA to avoid shade changes in sulfur dyes. Disodium phosphite is, however, a less effective curing catalyst than sodium hypophosphite. Monosodium phosphate is a fairly active catalyst and much less expensive than sodium hypophosphite, but it tends to produce a faint yellowing in cotton during heat curing.

Trask-Morrell and Andrews (1992) used the thermoanalytical technique to rank three inorganic salts of phosphorus-containing acids (i.e., monosodium phosphate, disodium phosphite, and sodium hypophosphite) as to their use with polycarboxylic acids in durable press finishing. Trask-Morrell and Andrews relied on three parameters to rank which catalyst was best with the acids. Rate of weight loss and percent residue, of acid alone, of the catalyst alone, and of the acid and the catalyst combined, were determined by the thermogravimetric method. Total heat of reaction was determined by differential scanning calorimetry. Trask-Morrell and Andrews found that a high percent residue, low rate of weight loss, and high total heat values corresponded to acids that provided good durable press properties in their fabric trials. According to their results on those three parameters, sodium hypophosphite is the best catalyst for BTCA, TCA and CA.

Fabric treatment with a solution containing BTCA and the catalyst sodium hypophosphite have shown good results in wrinkle recovery angle and durable press rating, but breaking strength and tear strength of finished fabrics were almost 50 per cent lower than those of untreated fabrics. Welch (1997) studied a three-component catalyst, consisting of triethanolamine, malic acid and sodium hypophosphite, to improve the strength and flex abrasion resistance of fabrics finished with BTCA. He found that the three-component catalyst system increased the breaking strength, tear strength, and flex abrasion resistance in the fabrics finished with BTCA. The flex abrasion resistance of the finished fabric was equal to that of untreated fabric, when the mole ratio of triethanolamine to the mixture of BTCA and malic acid was in the range of 0.90-1.00. The improvement of breaking and tear strength and flex abrasion resistance could be explained as follows: BTCA esterifies hydroxyl groups of triethanolamine and of malic acid to produce a graft copolymer having more flexible three-dimensional networks than the networks produced by BTCA and only the catalyst sodium hypophosphite.

BTCA, in the presence of sodium hypophosphite, provides the same level of durable press performance and better mechanical properties as found in DMDHEU treated cotton fabrics. Yet, the high cost of BTCA is an obstacle to mills' decisions to use BTCA as replacement for conventional durable press reactants. Brodmann (1990) evaluated durable press properties, shrinkage, physical performance, shade change of dyed fabrics, and yellowness of cotton fabrics treated with BTCA versus those parameters of DMDHEU finished fabrics. At the same add-on level, fabric finished with BTCA, in the presence of sodium hypophosphite, and fabric finished with DMDHEU had equally high durable press performance and low shrinkage properties. Tensile strength, tear strength and abrasion resistance of BTCA finished fabric were better than those of DMDHEU finished fabric. Fabrics finished with BTCA or with DMDHEU exhibited an equal degree of shade change when various classes of colorants were used on the fabrics; the equivalent shade changes occurred with vat, azo, and disperse dyes and with pigments, but not with sulfur dyes. The catalyst sodium hypophosphite has a high tendency to produce shade changes in sulfur dyes.

A research group at SRRC in New Orleans (Reinhardt, Bhattacharyya, Doshi, Sahasrabudhe & Mistry, 1994) evaluated the properties of cotton fabrics dyed with various direct dyes, reactant fixable dyes, and reactive dyes and then treated with two different types of crosslinking finishing agents, BTCA or DMDHEU. Durable press performance and strength properties of the BTCA treated cotton fabrics were equal to or better than those of the DMDHEU treated cotton fabrics. These results are similar to the results obtained by Brodmann (1990). The results supported that BTCA meets many of the requirements for satisfactory performance in terms of the level of reactivity, durable press properties, durability to laundering, fabric strength retention, fabric softness, freedom from formaldehyde, and absence of odor; however, the high cost of BTCA is an obstacle for practical applications.

According to the mechanism of the formation of ester crosslinks in cotton treated with a polycarboxylic acid, the lowest order of polycarboxylic acid

possible to form effective ester crosslinks in cellulosic fibers is tricarboxylic acid. Tricarboxylic acids of three types, 1,2,3-propanetricarboxylic acid (tricarballylic acid), 1,2,3-propenetricarboxylic acid (aconitic acid), and citric acid, have been examined for use in durable press finishes. Andrews and Trask-Morrell (1991) found that these tricarboxylic acids can be used to produce smooth drying properties in cotton fabrics.

Tricarballylic acid and aconitic acid provide a good level of durable press properties in fabrics treated with them, but aconitic acid produces severe discoloration in cotton fabric upon drying and curing. Therefore, aconitic acid cannot be used as a durable press finish for textiles. Citric acid also produces discoloration in treated fabrics when dried and cured at 180 °C for 90 seconds, but yellowing of citric acid treated fabrics is not as severe as that of aconitic acid treated fabrics. The discoloration problem caused by citric acid can be reduced or eliminated by certain aftertreatments, including washing (Welch & Andrews, 1989b, 1990). Tricarballylic acid does not discolor cotton fabric when the fabric is treated in the presence of sodium hypophosphite, and it provides good resiliency of treated cotton fabrics. Tricarballylic acid (TCA) is, however, of much less interest than BTCA because it requires a much higher percentage on weight of bath than does BTCA to achieve the same level of durable press performance and mechanical properties. BTCA and TCA are relatively expensive durable press finishes, so they are not available to use in large quantities for durable press finishing.

Welch and Peters (1997) studied two different mixtures of BTCA: the mixture of BTCA and malic acid, and the mixture of BTCA and maleic acid. With the mixture of BTCA and malic acid, the wrinkle recovery angle increased greatly from 244° to 272° in the fabric finished with 5.4% malic acid in the presence of 2-3% BTCA, but the breaking strength and tear strength of the finished fabric decreased with increasing BTCA concentration. The mixture of BTCA and maleic acid tended to cause precipitation of the monosodium maleate when 4-5% sodium hypophosphite monohydrate was added to the solution. Welch and Peters (1997) suggested that 1.5% phosphoric acid could redissolve this precipitate, but the treated bath had to be kept at 35 °C during the finishing process. A fabric finished with 1.7% BTCA and 6% maleic acid showed a higher durable press rating than that finished with only 6% maleic acid. The wrinkle recovery angle, breaking strength, and tear strength were not much affected in the presence of only BTCA.

The advantages of citric acid over others of the polycarboxylic acids are its low cost, proven lack of toxicity, and ready availability. It may be another candidate for an effective durable press finish. Andrews (1989, 1990) sought to improve the appearance properties of fabrics treated with citric acid and to minimize the associated yellowing problem. She found that fabric appearance, strength retention, and whiteness were improved when the catalyst to acid ratio was increased in the citric acid/sodium hypophosphite system. The whiteness index decreased as citric acid increased. Whiteness and smooth drying appearance levels were higher in the treatment with sodium hypophosphite as a catalyst than with other catalysts. Replacement of one-fourth of the citric acid

with BTCA in the presence of sodium hypophosphite provided good durable press properties, but those properties were not as good as with DMDHEU or BTCA based finishes. Therefore, citric acid may be useful as an extender for the expensive BTCA in applications that do not require extremely high durable press levels. Welch and Peters (1997) studied the effect of citric acid with BTCA on durable press performance. The results showed that as little as 0.25% BTCA with 7% citric acid imparted durable press performance in a finished fabric without decreasing the breaking strength and tear strength of the finished fabric. Not only can the addition of BTCA to citric acid improve durable press performance, but the addition of TCA to citric acid can do so as well. The addition of 1% TCA to 7% citric acid resulted in a durable press rating of 4.7, equal to that with 6.4% TCA by itself; 7% citric acid by itself produced only a 4.2 durable press rating.

Yang, Xu, Li, and Jiang (1998) studied the combination of citric acid with two polymers of maleic acids: homopolymer maleic acid (PMA) and terpolymer maleic acid (TPMA). These two polymers of maleic acid and citric acid have molecular structures similar to BTCA and can form five-membered cyclic anhydride intermediates. However, PMA and TPMA by themselves were less effective than BTCA because of the low mobility of the anhydride intermediates formed by PMA or TPMA to access the cellulosic hydroxyl during the curing process, while CA was less effective than BTCA due to its hydroxyl hindering its esterification with cotton cellulose. Combining either PMA or TPMA with CA may result in forming a polymer that could impart wrinkle resistance and good durable press performance to a finished fabric. Yang et al. found that the TPMA/CA combinations were more effective than the PMA/CA combinations for enhancing the wrinkle resistance of the finished fabrics. The nine fabrics finished with PMA/CA combinations had an average wrinkle recovery angle of 249°, while the nine fabrics finished with TPMA/CA combinations had an average of 261°. Within the studied ratios of either PMA or TPMA to CA, a 3:7 PMA or TPMA to CA ratio gave the highest wrinkle recovery angles in the finished fabric (268° for the fabric finished with PMA/CA and 276° for the fabric finished with TPMA/CA). Because TPMA/CA combinations provided better results in wrinkle recovery angles of the finished fabric than did PMA/CA, TPMA/CA combinations were chosen to study the effect of durability to washing. Yang et al. found that the fabric finished with 4:1 of TPMA/CA ratio had a durable press rating of 3.2 after 50 washing cycles, whereas the fabrics finished with BTCA and DMDHEU had durable press ratings of 3.2 and 3.3, respectively. The good wrinkle resistance and laundering durability of the fabrics finished with TPMA/CA combinations indicated that TPMA/CA combinations created a more efficient crosslinking system in the cotton fabrics than did TPMA or CA alone. However, the whiteness index of the fabric finished with the TPMA/CA was significantly lower than of those finished with BTCA or DMDHEU, because of the presence of CA in the TPMA/CA finish formulation and the high curing temperature of 185 °C.

Reinhardt, Bhattacharyya, Doshi, Sahasrabudhe and Mistry (1995) investigated the citric acid treatment of cotton fabrics dyed with direct dyes or with reactive dyes. They used triethanolamine (TEA) as an additive in the

treatment with citric acid in order to prevent fabric discoloration in the treatment. They compared the results in color strength and color difference of the citric acid treatment only and of the citric acid with the TEA system with those of the DMDHEU treatment. The DMDHEU treatment had less effect on color strength and color difference than did either of the citric acid treatment systems. The researchers could not conclude, however, that citric acid with TEA was a better treatment than citric acid in terms of color strength and color difference, because the color strength and color difference measured for the two different citric acid systems varied among the dyes.

Welch and Andrews (1989) and Welch (1994) summarized the processing conditions for each of several polycarboxylic acid crosslinkers that had given the best comparative results in fabric performance. As shown in Table 1, they compared the resultant durable press (DP) rating, wrinkle recovery angle (WRA), tear strength (TS) retention, and breaking strength (BS) retention, and they provided data on BTCA, TCA, and CA compared with those on DMDHEU. As noted in Table 1, similar DP ratings and WRA values resulted with the various crosslinkers when used at the concentrations and other processing conditions shown, but the tear and breaking strength retentions varied more and were lowest with DMDHEU crosslinking. Note, in the footnotes of Table 1, that one of the components of the finishing formulation is a fabric softener. Blanch (1995) explained that fabric softeners in the finishing formulations to achieve fabrics with wrinkle-free appearance also provide enhanced tear strength, abrasion resistance, and high-speed sewability. As well, the softeners are durable to laundering and have no discoloration effects on whites nor shade changes in dyed fabrics. A softening additive frequently used in finishing formulations is 1% polyethylene (Brodmann, 1990; Mehra & Mehra, 1991; Welch, 1994; Welch & Andrews, 1989 a&b).

Effective ester crosslinking reactions of polycarboxylic acids occur when each polycarboxylic acid contains at least three carboxyl groups on consecutive carbon atoms of its molecular chain or ring. An unsaturated dicarboxylic acid, such as maleic acid, can form a cyclic anhydride when heated due to the cis orientation of the two carboxyl groups in the acid compound; however, unsaturated dicarboxylic acids cannot form effective ester crosslinks like other polycarboxylic acids containing three or more carboxyl groups in each acid molecule. Welch and Choi (1992) suggested that the unsaturated dicarboxylic acids, such as maleic acid, can be copolymerized with other monomers in the presence of a free-radical initiator to yield more carboxylic groups, and thereby can be used as effective durable press finishing agents.

Choi (1992) and Welch and Choi (1992) investigated the effect of polymerization-crosslinking (PC) treatment with pad-dry-cure by using a mixture of maleic acid (MA) and itaconic acid (IA) on 3/1 cotton twill fabrics. Monomer concentration, pH, temperature and time of drying and curing, and initiator concentration were parameters examined in their research. Results showed that the wrinkle recovery angle was lower than that of the DMDHEU treated fabrics at concentrations of nine percent or more on weight of bath with a 1:1 ratio of MA to IA and sodium hypophosphite as a catalyst. Retention of tear strength and

Table 1 Textile properties resulting from certain polycarboxylic acid crosslinkers and DMDHEU on 100% cotton print cloth: durable press (DP) rating, wrinkle recovery angle (WRA), tear strength (TS) retention, and breaking strength (BS) retention

DP crosslinkers	DP rating	WRA	TS retention	BS retention
6.3 % BTCA[a]	4.5	300°	60%	54%
9.5% TCA[a]	4.6	300°	61%	57%
10.4% CA[a]	4.7	295°	54%	56%
6.0% DMDHEU[b]	4.6	303°	54%	44%

Note. [a]Processing conditions included 6.5% sodium hypophosphite monohydrate, 1% polyethylene emulsion (as a fabric softener), wet pick-up of 117-123 % o.w.f., predry at 85 °C for 5 min., and cure at 180 °C for 90 sec.
[b]Processing conditions included 1.5 % magnesium chloride hexahydrate, 1 % polyethylene emulsion, predry at 85 °C for 5 min., and cure at 160 °C for 3 min.

breaking strength decreased consistently when concentrations of the MA/IA mixture increased. The retention of tear and breaking strength of the fabric treated with the mixture of MA and IA was somewhat better than with DMDHEU.

In the drying temperature range of 90-110 °C, there was no statistically significant difference in the whiteness index of the treated fabrics; however, the whiteness index of the fabric treated with MA/IA was still lower than that of DMDHEU treated fabrics. The drying temperature of 100 °C for 10 min. was the best condition to achieve good balancing of durable press properties and mechanical properties for the MA/IA treated fabrics. The effects of curing temperature and time on durable press rating and wrinkle recovery angle of the treated fabrics depended on the combination of curing time and temperature used for finishing. Whiteness index continuously decreased when curing temperature increased. The curing temperature at 180 °C for 90 seconds was the best combination to provide both good durable press rating and good wrinkle recovery angle.

Various free-radical initiators, such as azo initiator, hydrogen peroxide, potassium persulfate, and sodium persulfate, were used with the MA/IA mixture for treating fabrics. The fabric treated with potassium persulfate initiator had higher wrinkle recovery angle and durable press rating than did the fabrics treated with other initiators. The researchers found that pH of the treating bath of the MA/IA mixture should be kept between 1.7 and 4.0 for effective esterification and crosslinking with cellulose hydroxyl groups.

Choi (1992) studied the MA/IA mixture via PC treatment with the pad-dry-cure technique. He concluded that good DP rating between 4 and 4.5 and wrinkle recovery angle of about 270 to 290 degrees on 3/1 twill cotton fabrics resulted from treatment in a system containing 8.9-15.6 % o.w.f. of a mixture at 1:1 mole ratio of MA to IA, along with sodium hypophosphite as a catalyst at 1:1 or 1:1.33 mole ratio of monomers to catalyst at 100 °C drying temperature for 10 minutes, and at 180 °C curing temperature for 2 minutes. Those results were comparable to those of the conventional DMDHEU treated fabrics.

Application Techniques for Durable Press Finishing

Crosslinking agents can be applied to cotton fabrics in a number of ways. The pad-dry-cure technique and the wet-fixation technique are the most common ways to apply durable press finishes on cotton fabrics.

Pad-Dry-Cure Technique

Pad-dry-cure is the conventional technique for applying finishing agents to cotton fabrics. It is a simple and easy technique. The fabric is immersed, for 5-10 minutes, in the aqueous treating solution containing the durable press finish, curing catalyst, fabric softener, wetting agent, and water. Then the fabric is padded through squeeze rolls to give a specified wet pick-up reported as percent on weight of fabric (% o.w.f.). After that, the fabric is dried and cured for a specified time at a specified temperature. The pad-dry-cure technique results in finished fabrics with good wrinkle recovery angles and DP ratings, but with high losses in tensile and tear strength and abrasion resistance. The problems occur with both formaldehyde reactants and nonformaldehyde reactants used as DP

finishes. Shin, Hollies, and Yeh (1989) noted two major factors contributing to the loss in mechanical properties: fiber degradation caused by the acid catalyst at elevated temperatures; and the restriction of stress distribution within the fibers due to their rigid crosslinking by monomeric resins. Hollies and Getchell (1967) developed the wet fixation process for improving the retention of mechanical properties in finished fabrics.

Wet-Fixation Technique

Hollies and Getchell (1967) suggested that the use of two resin components is a key to balancing good strength properties and high levels of DP performance in the finished fabric. The first component, called a polymer builder, polymerizes with itself under the treatment conditions and keeps the fibers in swollen state during treatment. This step is carried out in the wet state. The second component is called a crosslinking agent. It contributes only moderately to fiber swelling when wet fixed with a polymer builder. It can enhance wrinkle recovery in the fabrics once catalyzed and dried. According to the wet fixation process reported by Hollies and Getchell (1967), cotton fabric was padded through the polymer builder, with mineral acid, at 75% to 100% wet pick-up. The wet fabric was then wrapped in a Mylar envelope (polyethylene bag now used instead), and then placed in an oven at a specified condition. In this stage, no moisture was allowed to escape from the envelope. The fibers swelled and permitted the resins to enter; the resins polymerized to some extent in the fibers in the presence of the mineral acid. Upon removal from the envelope, the fabric was neutralized in sodium carbonate solution, and then washed and tumble dried. Subsequently, the fabric was padded with a solution containing crosslinkers, catalysts, and other additives, such as softener and wetting agent. Then the treated fabric was dried and cured. Crosslinking agent can be either applied by the pad-dry-cure technique after the wet fixation of the polymer builder, or applied simultaneously with the polymer builder before the wet fixation process. At least 8% to 12% of resin add-on must be fixed in the fabric to achieve the enhancement of fabric performance.

The main way that the wet fixation process differs from the conventional pad-dry-cure technique is the step in which the resin impregnated fabric is held in the moist state for a certain time at a certain temperature. This step allows for resin penetration, deposition, and polymerization inside the fibers.

Hollies (1967) described the morphological changes in cotton fibers exposed to the wet fixation process and how the changes related to altered mechanical properties of the treated fabrics. He found that the fibers in the pad-dry-cure treated fabric were more collapsed than those in the wet fixed samples. He revealed that the wet fixation crosslinking resin resided differently in cotton fibers after wet fixation than after only the conventional pad-dry-cure, in three respects: (a) wet fixed samples had resin penetrated into the fabric, yarn, and fibers, whereas pad-dry-cure samples had substantial resin deposited on the fiber surface only; (b) wet fixed samples were less effective in influencing the light speed across a fiber cross-section, which was interpreted as an intimate mingling of resin with the cellulose structure and a corresponding loss in birefringence; and (c) the fibers in wet fixed samples showed a substantial

increase in refractive index after the curing step, which may have been due to a chemical reaction with cellulose beyond crosslinking. Hollies suggested that this last respect may be related to the basic changes in mechanical properties of fabrics treated by the wet fixation process.

Other researchers (Bertoniere, Blouin, Martin & Rowland, 1974; Rowland, Bertoniere & Martin, 1974; Shin, Hollies & Yeh, 1989) have posited that the high level of DP properties, along with retention of mechanical properties, in cotton fabrics treated by polymerization-crosslinking used with the wet fixation process are the consequence of two phenomena. One is the deconvolution of cotton fiber as a result of the deposition of polymerizing resins within the interior of wet swollen fibers, thereby releasing internal strain in the fiber. The other phenomenon is the plasticity of interfaces between microstructural units of cellulose as a result of the formation of long, flexible crosslinks of cellulose through the functional groups in the resins.

Shin, Hollies, and Yeh (1989) investigated the polymerization-crosslinking treatment using mixed resin systems containing a polymerization resin and a crosslinker. They used N-methylolacrylamide as a polymerizing resin, and DMDHEU or urea-formaldehyde (UF) as crosslinkers. The nonconventional treatment techniques of wet fixation and steam fixation were used for their study of the PC treatment of cotton fabrics. Their results showed that the PC treatment, with either wet fixation or steam fixation, provided the treated samples with an overall durable press performance superior to those treated with DMDHEU. The conventional pad-dry-cure process did not result in significantly changing the morphological structure of the fibers because of poor penetration of monomers. The researchers suggested that the penetration of the polymerization resin into the fiber interior within the treated fabrics using nonconventional processes was responsible for the improved performance properties. The disadvantage of the wet-fixation process is that it is time consuming due to the step called fixation, where a treated fabric is fixed and sealed in a polyethylene bag at a specified temperature for a specified time. If the treated fabric is allowed to fix at room temperature, the time required for fixation is many hours. Reducing the time of fixation can be done by placing the treated fabric in an oven at a temperature higher than room temperature.

Shin et al. (1989) mentioned that improvement of mechanical properties by the PC treatment is dependent on the control of three key processes: the diffusion of monomers into the fiber interior; polymerization of monomers within the fibers; and condensation crosslinking or anchoring of polymers to the fibers. The monomer diffusion process is more complex than the others. This process is expected to depend on temperature, type of monomers including size and shape, polymerization initiator, and the size and total volume of pores in the fibers. These factors affect the changes in fiber structure and thus the changes in fabric performance properties, such as strength, abrasion resistance, wrinkle recovery angle, and bending stiffness.

PC treatment with the pad-dry-cure process can be done by adding an initiator to speed up a reaction to occur within 10-20 minutes. PC treatment normally employs formaldehyde based monomers such as melamine and N-

methylolacrylamide which release formaldehyde. Little has been done to examine alternative PC treatments that do not generate formaldehyde vapors.

Evaluation of the Properties of Durable Press Cotton Fabrics

To accomplish a perfect durable press finishing of cotton fabrics is to employ a finish formulation that will obtain the desirable balance of physical properties and durable press performance (Turner, 1994). If a finish formulation gives an excessive crosslinking network in a finished fabric, the strength and abrasion resistance may be too low for adequate wear life for the finished fabric. On the other hand, if a finish formulation gives too little performance on crosslinking networks, the durable press performance may be inadequate to provide good wrinkle resistance of the finished fabric. Therefore, the evaluation of the properties of durable press finished cotton fabrics covers the testing of seven properties: tensile strength, tear strength, durable press rating, wrinkle recovery angle, abrasion resistance, color strength and shade changes, and wash resistance of cuffs after repeating home laundering and tumble drying for 5, 10, and 25 times (Ryan, 1971; Turner, 1994). The testing methods for evaluating the properties of durable press cotton fabrics follow the standard testing methods contained in the yearly technical manuals of the American Association of Textile Chemists and Colorists (AATCC) or of the American Society for Testing and Materials (ASTM), which are commonly used in industry and in development laboratories. Further discussion of evaluation procedures is in the Procedure chapter.

Summary of Literature Review

Polycarboxylic acids have been developed for use in new durable press finishes for cotton fabrics to replace the N-methylol compounds, especially the conventional DMDHEU reactant, because of increasing concern about toxicity of formaldehyde vapors from the N-methylol compounds. Polycarboxylic acids react with cellulosic fibers to form ester-linkage crosslinked networks, which impart durable press properties. The mechanism of cellulose esterification by a polycarboxylic acid involves the formation of a cyclic anhydride intermediate by the dehydration of two carboxyl groups, and the reaction between the cyclic anhydride intermediate with a hydroxyl group of cellulose to form an ester linkage (Andrews & Trask-Morrell, 1991; Welch, 1994). The formation of effective ester-linkage crosslinked networks in cotton cellulose, to achieve good durable press properties, requires at least three carboxylic acid groups in each polycarboxylic acid molecule. Both BTCA and CA, each having at least three carboxylic groups, can serve as effective crosslinking agents.

Among the polycarboxylic acids that have been studied, 1,2,3,4-butanetetracarboxylic acid (BTCA) is the most effective crosslinking agent for cotton cellulose. BTCA, in the presence of sodium hypophosphite, provides the same level of durable press properties and finish durability in laundering as does the DMDHEU reactant. Although BTCA can be an effective durable press finish for cotton fabrics, without releasing formaldehyde vapors, the high cost of BTCA is an obstacle to its wide use in the textile industry today. Citric acid (CA) is

another candidate to replace DMDHEU because of its low cost, lack of toxicity, and ready availability. CA, in the presence of sodium hypophosphite, does not provide as good a level of durable press properties as does DMDHEU reactant.

Unsaturated dicarboxylic acids, such as maleic acid, cannot form effective ester crosslinks like BTCA or CA. Polymerization of a free radical of unsaturated dicarboxylic acid with other monomers could provide a new polycarboxylic acid formed by two different monomers to be used as a durable press reactant. The mixture of maleic acid and itaconic acid by polymerization-crosslinking treatment with pad-dry-cure, in the presence of a catalyst, provides wrinkle recovery angles and durable press ratings close to those from DMDHEU (Choi, 1992; Choi & Welch, 1992). In recent years, experimentation with combinations of BTCA and other cheaper polycarboxylic acids provides the prospect of decreasing the amount and cost of BTCA used.

Polycarboxylic acids solve the problem of formaldehyde release. Yet, the high cost of BTCA and the loss of mechanical properties of cotton fabrics with polycarboxylic acid reactants suggest the need for further study. Polymerization-crosslinking with wet-fixation uses a combination of two resin components, a polymer builder and a crosslinker, which could improve breaking and abrasion resistance at a high level of durable press properties because it allows for resin penetration, deposition, and polymerization inside the fibers. Shin, Hollies, and Yeh (1989) investigated the polymerization-crosslinking treatment, with wet fixation or steam fixation, of a mixture of two resin components: N-methylolacrylamide as a polymer builder, and DMDHEU or urea-formaldehyde as a crosslinker. They found that the polymerization-crosslinking treatment, with either wet fixation or steam fixation, provides the treated samples with an overall durable press performance superior to those treated with conventional DMDHEU. Both of the above procedures require specific equipment for the fixation step. An oven is needed in the wet fixation process, and a steam chamber is needed in the steam fixation process for the fabric fixation. After the fabric fixation in those processes, the fabric which is finished with durable press crosslinkers must be dried and cured by running through a tenter machine. The disadvantages of the wet fixation and the steam fixation processes are that they are time and energy consuming and they require more equipment for the fixation step. In comparison, fabric finishing with the pad-dry-cure process can be done continuously from finishing, drying, and curing in one tenter machine.

The concept of polymerization-crosslinking treatment with polycarboxylic acid reactants has not been investigated much. Moreover, a new research area in wrinkle resistance is the use of various new combinations of unsaturated carboxylic acids, such as acrylic acid, maleic acid, itaconic acid, or mixtures of any two unsaturated carboxylic acids, and other polycarboxylic acids having more than two carboxylic groups per molecule, such as BTCA or CA.

CHAPTER III
Setting of the Problem

Garments made from 100% cotton fabrics are comfortable because they have good absorption ability; however, cotton fabrics have several drawbacks. They wrinkle badly in washing and tumble drying and during wearing; therefore, they need to be ironed before wearing again. In addition, they tend to shrink when washed. Cellulose chains can slip under moist conditions and, on their own, will not return to their original position. This phenomenon is responsible for wrinkling in cotton fabrics (Andrews, 1992, 1995; Cooke & Weigmann, 1982 a&b; Smith & Block, 1982 a&b). The drawbacks of cotton fabrics can be ameliorated by properly applying durable press finishes that cause crosslinking between adjacent cellulose chains in treated cotton fibers. The hydrogen bonds and van der Waals forces between cellulose chains are not sufficient to prevent slippage of adjacent chains in untreated cotton fibers, but the crosslinking can reduce the slippage within the fibers of treated cotton fabrics.

The crosslinking agents now used in the commercial finishing of cotton fabrics are the N-hydroxymethyl and N-alkoxymethyl compounds of urea, cyclic urea, carbamates, and aminotriazines. These crosslinking agents are classified as formaldehyde reactants (Cooke & Weigmann, 1982 a&b; Peterson, 1987). The most popular formaldehyde reactant used in textile finishing is DMDHEU. It provides good durable press properties, is resistant to hydrolysis, and releases formaldehyde vapors much less than do other formaldehyde reactants. Formaldehyde is listed as a hazardous and toxic substance by the Occupational Safety and Health Administration (OSHA). The OSHA has categorized formaldehyde as a probable human carcinogen (Andrews, 1995). Several studies by the Chemical Industry Institute of Toxicology show that formaldehyde is a carcinogen to animals. To humans, it is a severe eye irritant, a mucous membrane irritant, a skin irritant and sensitizer, and it is toxic if ingested (Cooke & Weigmann, 1982a). Because of the concern about formaldehyde hazards to workers in the textile industry and also to consumers, formaldehyde-free crosslinking agents for producing durable press properties are of interest to replace DMDHEU.

N, N' dimethyldihydroxyethyleneurea (DMeDHEU) and 1,3-dimethyl-4,5-dihydroxyethyleneurea (DHDMI) are two of the nonformaldehyde reactants that researchers are investigating for durable press finishing. These two reactants can react with cellulose through the pendant hydroxyl groups on the rings; however, the durable press properties obtained with either reactant do not equal those obtained with DMDHEU (Andrews, 1995; Cooke & Weigmann, 1982a).

Researchers at the Southern Regional Research Center (SRRC), New Orleans, LA, have been vigorously developing new and modified resins, with the proper catalysts, that can serve as formaldehyde-free durable press agents (Welch, 1988). Polycarboxylic acids (PCA) are nonformaldehyde reactants that are possible replacements for the conventional finishing reactant. The main advantages of PCA are that they are formaldehyde-free, do not have a bad odor, and produce a very soft fabric hand. A PCA reacts with the hydroxyl groups of

cellulose and forms the ester linkage with cellulose, rather than the ether linkage as do the N-hydroxymethyl and N-alkoxymethyl compounds.

Butanetetracarboxylic acid (BTCA) and citric acid (CA) reactants are the most important of the PCA for the textile industry today. The best results in DP finishes with PCA have been obtained with BTCA in the presence of sodium hypophosphite as the catalyst (Welch; 1988; Welch & Andrews, 1989 a&b). BTCA, in the presence of sodium hypophosphite, provides durability similar to that from DMDHEU, but the high cost of BTCA is an obstacle to mills' decisions to replace the DMDHEU reactant with BTCA.

Citric acid is another candidate to replace DMDHEU. The advantages of CA over other PCA are low cost, proven lack of toxicity, and ready availability (Andrews, 1990). Finishes from citric acid exhibit the same level of durable press performance as that from DHDMI, but not the same level of durable press appearance properties as from DMDHEU. Citric acid, in the presence of an inappropriate catalyst, causes yellowing of treated cotton fabrics (Andrews & Trask-Morrell, 1991). Smooth-drying properties can be achieved with 1,2,3-propanetricarboxylic acid (TCA), but good DP performance depends on using a high percentage on weight of bath (% o.w.b). In addition, the price of TCA is higher than that of DMDHEU (Andrews, 1990; Andrews & Trask-Morrell, 1991).

As mentioned, cost and durable press performance of the PCA reactants are the two main drawbacks to using the PCA instead of the conventional DMDHEU reactant. Some PCA yield good durable press properties, but are prohibitively expensive; others are sufficiently economical, but yield inadequate durable press performance. The major loss of mechanical properties of treated cotton fabrics with PCA reactants is another problem.

Some researchers (Bertoniere, Blouin, Martin & Rowland, 1974; Choi, 1992; Hollies & Getchell, 1967; Rowland, Bertoniere & Martin, 1974; Shin, Hollies & Yeh, 1989) proposed that the use of the polymerization-crosslinking (PC) treatment could improve breaking strength and abrasion resistance at a high level of DP properties compared to those of fabric finished with conventional DMDHEU. This treatment has been used with a wet fixation process and containing two resin components, a polymer builder such as N-methylolacrylamide and a crosslinking agent such as urea formaldehyde.

A way to reduce the expense of BTCA is to use a mixture of BTCA with cheaper carboxylic acids, such as acrylic acid, maleic acid or itaconic acid. Polycarboxylic acids having three or more carboxyl groups, when used with an appropriate catalyst, can provide a high degree of wrinkle resistance as well as smooth drying properties (Andrews, Welch & Trask-Morrell, 1989; Welch, 1990; Welch & Andrews, 1989 a&b, 1990). Maleic acid and itaconic acid are unsaturated dicarboxylic acids having two carboxyl groups, so they cannot provide effective crosslinking reactions with cellulose to achieve a high level of DP performance. Copolymerization of unsaturated carboxylic acid with other monomers in the presence of a free-radical initiator, such as potassium persulfate or hydrogen peroxide, could provide more carboxyl groups in the acid molecule (Choi, 1992; Choi & Welch, 1992). After copolymerization, modified unsaturated carboxylic acids would contain more than two carboxyl groups in

their structure molecules. Such a polymer might be used as an effective DP finishing agent.

In PC treatment to balance the mechanical and durable press properties of treated fabrics, maleic acid, itaconic acid and acrylic acid can be used as polymer builders, and BTCA or CA can be used as crosslinking agents. The combination of PCA reactants under the PC treatment with pad-dry-cure may provide a new durable press finish which reacts with cellulose and forms effective ester crosslinks to impart durable press properties and provide good mechanical properties in treated fabrics. The polymerization-crosslinking treatment with pad-dry-cure could be done by adding an initiator to promote a faster polymerization reaction. In addition, the pad-dry-cure process is the simplest and easiest method to achieve wrinkle resistance. PC treatment with pad-dry-cure may be a method for achieving a balance of durable press and mechanical properties when used with PCA reactant combinations.

Theoretical Framework

Polymer chain slippage within cellulosic fibers under moist conditions is responsible for wrinkling in cotton fabrics. Logically, crosslinking adjacent cellulose chains should be a way of improving wrinkle resistance. Research and industrial practice have shown that the crosslinking of cellulose chains with appropriate chemical reactants can improve the durable press (DP) properties of cotton fabrics.

N-methylol groups can react with cellulose hydroxyl groups, or N-methylol groups can react with -NH groups to form three-dimensional polymers. Among N-methylol compounds, dihydroxymethyl-4,5-dihydroxyethyleneurea (DMDHEU) is the most common crosslinking agent for DP finishes on cotton or cotton-blend fabrics. Cotton fabric treated with DMDHEU has a better rating of DP appearance and a higher wrinkle recovery angle than does untreated cotton fabric; however, this treatment reduces the breaking strength and tear strength of treated cotton fabric as compared to untreated cotton fabric (Andrews, 1990; Andrews, Welch & Trask-Morrell, 1989; Choi & Welch, 1992; Welch & Andrews, 1989a).

Both the improvement in DP properties and the deterioration in mechanical properties of fabrics are directly proportional to the degree of crosslinking (i.e., the number of crosslinks imparted in the fibers). The percent add-on of a crosslinking reagent applied to the fabric and the degree of curing determine the number of crosslinks. The degree of curing refers to the percentage of the applied chemical reactant that actually forms crosslinks with cellulose. The degree of curing is influenced by the chemical structures of reactants, type of catalyst, ratio of catalyst to reactant, and duration and temperature of cure.

The greater the amount of reagent, on percent weight of bath (% o.w.b.), applied to the fabric, the greater the chances of crosslinking occurring; however, consideration must be given to the cost of using reactants in DP finishing and the

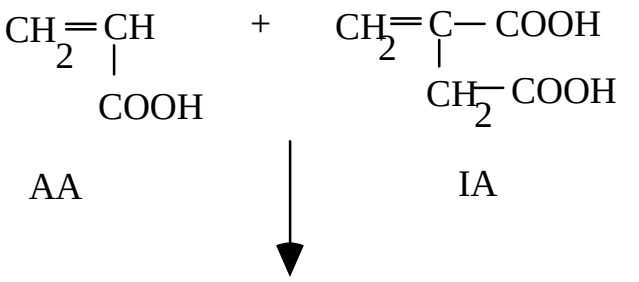
relationship of the % o.w.b. of reactants to the yield of DP performance obtained and to the deterioration in mechanical properties in treated fabric.

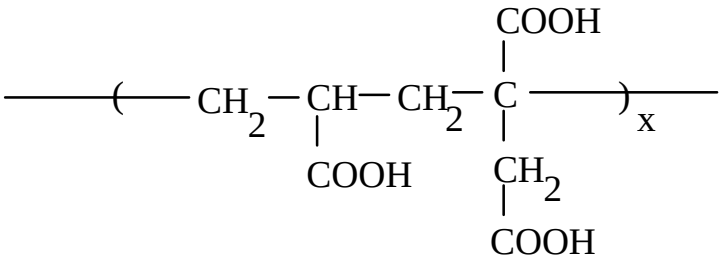
Reactants used in DP finishing can be categorized as formaldehyde or nonformaldehyde reactants (Cooke & Weigmann 1982a; Peterson, 1987). Because of potential hazards to workers in the textile industry and to consumers, as a result of the release of formaldehyde vapors by formaldehyde reactants during DP finishing, storage, and consumer use, there is interest in replacing the DMDHEU reactant with a nonformaldehyde reactant. Most of the discussion here focuses on formaldehyde-free crosslinking agents.

Scientists at the Southern Regional Research Center in New Orleans, LA, have studied polycarboxylic acids as nonformaldehyde alternatives for DP finishes on cotton fabric (Andrews, Welch & Trask-Morrell, 1989; Welch, 1990; Welch & Andrews, 1989 a&b, 1990). They found that a high degree of wrinkle resistance as well as smooth drying properties result from the use of polycarboxylic acids (PCA) having three or more carboxyl groups, in the presence of alkali metal salts of a phosphorus-containing acid (i.e., sodium hypophosphite, disodium phosphite, or monosodium phosphate). According to those scientists, PCA, in the presence of the alkali metal salts of inorganic phosphorous-containing acids, react with cellulose via an anhydride mechanism. In the presence of heat and catalyst, two carboxylic groups form a five-membered anhydride ring and release one molecule of water. The cyclic anhydride reacts with cellulose to form an ester and regenerate one carboxylic acid group. Therefore, each polycarboxylic acid molecule must have at least three carboxylic acid groups to provide effective crosslinks with cellulose. BTCA, TCA and CA, each having at least three carboxylic groups, can serve as effective crosslinking agents. Polycarboxylic acids, especially BTCA, provide good durable press performance and finish durability in laundering, and they solve the problem of formaldehyde release; however, the loss of mechanical properties in cotton fabric treated with PCA reactants needs to be solved.

Researchers (Choi, 1992; Shin, Hollies & Yeh, 1989) have suggested that two major factors account for the general problem of poor mechanical properties in DP finished cotton fabrics. The two factors are the fiber degradation caused by acid catalyst at elevated temperature and the restriction of stress distribution within the fibers due to their rigid crosslinking by chemical reactants. Other researchers (Bertoniere, Blouin, Martin & Rowland, 1974; Rowland, Bertoniere & Martin, 1974; Shin et al., 1989) have posited that two phenomena account for the high levels of DP properties, along with retention of mechanical properties, in cotton fabrics treated by polymerization-crosslinking used with the wet fixation process. One phenomenon is the deconvolution of cotton fibers as a result of the deposition of polymerization resins within the interior of wet swollen fibers, with the release of internal strain in the fibers. The other phenomenon is the plasticity of interfaces between microstructural units of cellulose as a result of the formation of long, flexible crosslinks with cellulose through the functional groups in the resins. Polymerization of a mixture of IA/AA could provide a long chain polymer that has enough active functional groups to react with hydroxyl groups within cotton fibers and form flexible crosslinks.

The present research examines the mechanical and DP properties of cotton fabric treated by polymerization-crosslinking with pad-dry-cure and with BTCA/IA/AA combinations. A mixture of IA/AA, in the presence of potassium persulfate, may polymerize to have more than three carboxyl groups in the acid structure. Such a polymer might be used as an effective DP finishing agent. The polymerization structure resulting from the mixture of IA/AA is shown below.





It may be possible that polymerization-crosslinking, used with pad-dry-cure, of a BTCA/IA/AA combination and an appropriate initiator and curing catalyst could yield DP properties similar to those provided by BTCA alone. After polymerization-crosslinking with pad-dry-cure, a new chemical reactant formed by the mixture of the two resin components may provide a chemical structure having enough carboxyl groups in the right position to form ester linkages with cellulose. Moreover, if the new chemical reactant has a chain long enough to provide highly flexible crosslinks in cotton fibers, the treated fabrics may perform well both in wrinkle resistance and strength retention. A polymerization-crosslinking process involving reducing agents, such as potassium persulfate, should normally be effected in an inert atmosphere, such as atmospheric nitrogen, to avoid the inhibition of polymerization by oxygen. The duration time of PC treatment with pad-dry-cure is much shorter than that in PC treatment with wet fixation, so the effect of the oxygen scavenger on the polymerization reaction may be less severe with pad-dry-cure. According to the results of preliminary work in this study (in Chapter VI), performing the polymerization-crosslinking process under atmospheric nitrogen did not provide better results in wrinkle recovery angle and breaking strength retention of the finished fabrics than did performing the process in the ambient condition.

Several catalysts for PCA have been reported to promote the crosslinking reaction. Selection of the proper catalyst is important for two reasons: (a) a stronger catalyst is required with reactants of low reactivity to promote reaction;

and (b) the catalyst may affect the mechanical and durable press properties and the discoloration of treated cotton fabrics. Sodium hypophosphite is the most effective catalyst for promoting DP properties, but it is expensive and it discolors some sulfur dyed fabrics. Disodium phosphite does not produce shade changes in dyed fabrics, and it is inexpensive; however, it is a less effective curing catalyst than sodium hypophosphite. Monosodium phosphate is a fairly active catalyst and is inexpensive, but it tends to cause discoloration on white fabric at high temperature curing. Sodium hypophosphite, which is the most effective catalyst for PCA, is used as the catalyst in this research.

The ratio of catalyst to resin should be appropriate to promote the optimal degree of curing for the best DP properties on treated cotton fabrics (Choi, 1992; Welch & Andrews, 1989b, 1990). In curing, the time should be long enough and the temperature high enough to allow the crosslinking reaction to occur maximally. Welch (1990) suggested that curing for 45-90 seconds at 180 °C was necessary for obtaining a balance of mechanical properties, DP performance, and durability of PCA finishes.

Conceptual Definitions

Crosslinking: "the stabilization of cellulosic fibers through chemical reaction with certain compounds in such a way that the cellulose chains are bridged across or crosslinked" (Celanese, 1978, p.37).

Degree of crosslinking: the number of crosslinks in cellulosic fibers, reported as a percentage of insoluble cellulose (Bullock et al., 1966).

Polymerization-crosslinking treatment: a treatment involving two components, polymer builder and crosslinker, which is performed to balance good mechanical properties and good durable press performance of the finished fabrics (Hollies & Getchell, 1967).

Polymer builder: a chemical reactant containing unsaturated bonds that can polymerize with itself or with other reactants, and that can keep the fibers in swollen state under DP finishing conditions (Hollies & Getchell, 1967).

Crosslinker: a chemical reactant containing more than two functional groups that are capable of reacting with cellulose to form crosslinking networks in cellulosic fibers (Hollies & Getchell, 1967).

Pad-dry-cure technique: a conventional application process in durable press finishing that involves (a) padding, where fabric is impregnated in a pad bath and then padded through squeeze rolls, and (b) subsequent drying and curing at a specified temperature and time in an oven (Hollies & Getchell, 1967; Shin, Hollies & Yeh, 1989).

Wet fixation technique: an application process in durable press finishing that is similar to the pad-dry-cure technique except that a fixation step follows padding wherein treated fabrics are sealed in polyethylene bags and placed in an oven at a specified temperature and time; fabrics then are dried and cured at a specified temperature and time as in the pad-dry-cure technique (Hollies & Getchell, 1967; Shin, Hollies & Yeh, 1989).

Durable press: the property of a fabric that is "the ability to retain substantially its initial shape, flat seams, pressed-in creases, and unwrinkled

appearance during use and after laundering or drycleaning" (AATCC, 1991, p. 207).

Wrinkle recovery: " that property of a fabric which enables it to recover from folding deformations" (Celanese, 1978, p.160).

Breaking strength: the maximum load required to break or rupture a fabric specimen in the vertical direction (ASTM, 1995).

Tearing strength: the average force required to continue a tear previously started in a fabric specimen (ASTM, 1994).

Whiteness index: the number refers to how white a fabric specimen is compared with a standard white material (AATCC, 1991, p. 161).

A Listing of Acronyms

AA: acrylic acid
BS: breaking strength
BTCA: 1,2,3,4 butanetetracarboxylic acid
CA: citric acid
DMDHEU: dihydroxymethyl-4-5-dihydroxyethyleneurea
DP: durable press
IA: itaconic acid
MA: maleic acid
PC: polymerization-crosslinking
PCA: polycarboxylic acids
PMA: homopolymer of maleic acid
TCA: 1,2,3 propanetricarboxylic acid
TPMA: terpolymer of maleic acid
TS: tear strength
WRA: wrinkle recovery angle

Purpose and Objectives

The purpose of this research was to examine the mechanical and DP properties of cotton 3/1 twill woven fabric treated by a polymerization-crosslinking process with pad-dry-cure, using BTCA/IA/AA combinations; the mixture of IA/AA was used as a polymer builder and BTCA was used as a crosslinker. According to the results of the preliminary analysis (see details in Chapter VI), BTCA/IA/AA combinations provided better results in wrinkle recovery angles of the finished fabric than the other chemicals did. Therefore, the BTCA/IA/AA was studied further in the main research. The results on measured fabric properties were analyzed to assess the optimal treatment conditions for obtaining a balance of the mechanical and DP properties.

The objectives were to determine, under the treatment conditions used in the research, the effects of the several DP finishing variables on key properties of 3/1 twill finished cotton fabric: the mechanical properties of tear strength and breaking strength, the durable press properties measured by durable press rating and wrinkle recovery angle, and the whiteness. The durable press finishing variables examined were (a) two concentrations, 2% and 3%, of BTCA, (b) two concentrations, 6.4% and 9.6%, of IA, (c) two concentrations, 1.77% and

3.54%, of AA; (d) two mole ratios, 1:1 and 1:0.8, of the monomers to the catalyst sodium hypophosphite monohydrate; and (e) two curing times, 90 sec. and 3 min., at 180 °C. The results for the fabric specimens treated by the polymerization-crosslinking, using pad-dry-cure with BTCA/IA/AA combinations, were compared with the results on the same parameters for fabric specimens treated with the conventional DMDHEU treatment and the 6.3% BTCA treatment, with pad-dry-cure. The DMDHEU treatment was with 12% DMDHEU, 1.5% magnesium chloride hexahydrate, 1% polyolefin emulsion, and 0.2% Triton X-100, followed by drying at 100 °C for 5 minutes and curing at 160 °C for 3 min. The BTCA based finish treatment was with 6.3% BTCA, 6.5% sodium hypophosphite monohydrate, 1% polyolefin emulsion, and 0.2% Triton X-100, followed by drying at 85 °C for 5 min. and curing at 180 °C for 90 sec.

The amount of initiator and the temperature and time of drying at 100 °C for 10 min. were fixed influence factors in the research. The initiator, potassium persulfate, in the amount of 1.5% on weight of monomers (% o.w.m.) was used, as Choi (1992) found this was sufficient to polymerize the mixture of MA/IA to impart good durable press properties. Choi also found that drying at 100 °C for 10 min. provided a durable press rating and wrinkle recovery angle close to those obtained with conventional DMDHEU treated cotton fabric. The amount of 1.5% o.w.m. of potassium persulfate and 10 min. drying at 100 °C were two fixed influence factors used in the preliminary work. Based on the measurement results on breaking strength and wrinkle recovery angle from the preliminary work, the fabric treated with the BTCA/IA/AA combinations had good results on both properties that were close to those obtained from BTCA only. This amount of potassium persulfate used in preliminary work was enough to drive the reaction of BTCA/IA/AA combinations to occur in a short polymerization-crosslinking time with pad-dry-cure; therefore, 1.5% o.w.m. of potassium persulfate and 10 min. drying at 100 °C were chosen for this research.

Research Hypotheses Associated with Each Objective

Objective

This research sought to determine the effect of varying concentrations of BTCA/IA/AA combinations, the effect of different mole ratios of the monomers to sodium hypophosphite, and the effect of the curing time on the mechanical, durable press, and whiteness properties of finished cotton fabric.

Rationale for associated hypotheses. A key to balancing good strength and good durable press performance is the use of two resin components, a polymer builder and a crosslinker (Bertoniere et al., 1974; Hollies & Getchell, 1967; Rowland et al., 1974; Shin et al., 1989). The polymer builder polymerizes with itself and holds the fibers in a swollen state. In this swollen state, the convolutions of cotton fibers become altered from their collapsed state so polymerizing resins can deposit within the interior of the wet swollen fibers. This phenomenon can release the internal strain of the fiber. The IA/AA mixture may polymerize and thereby form a polycarboxylic acid containing more than two carboxyl groups which differs in structure from either original chemical structure of the IA or AA. Welch (1988) explained that the cellulose crosslinking reaction

with BTCA involves the formation of a cyclic acid anhydride which reacts with cellulose hydroxyl groups to form an ester-type linkage connecting adjacent cellulose chains in a three-dimensional network inside cellulosic fibers. It is the network crosslinking that imparts wrinkle recovery, shrinkage control, and smoothness to cotton fabrics.

In polymerization-crosslinking, the BTCA and IA/AA mixture may form a cyclic acid anhydride that reacts with cellulose chains, or either the BTCA or the IA/AA mixture may form a cyclic anhydride by itself and react with cellulose chains directly. The first case will result in more flexibility among microstructural units of cellulose due to longer chain formations, so the mechanical properties can be improved over those in the latter case. The relative concentrations of monomers is one factor affecting the polymer chain formed by the polymerization-crosslinking process. Generally, higher concentrations of monomers may drive a reaction forward at a faster rate than can lower concentrations of monomers. Each reactant in the BTCA/IA/AA combination may or may not affect the fabric properties by itself. The BTCA and the mixture of IA/AA may enhance or deteriorate the ability of BTCA or the ability of IA or AA in the IA/AA mixture to act on the mechanical and durable press properties and whiteness of finished cotton fabrics. The contents of the IA/AA mixture may depend on each other to promote their effective polymerization to obtain more carboxyl groups that are able to react effectively with the cellulose hydroxyl groups of cotton. The BTCA/IA/AA combination may not form effective ester linkages with cellulose hydroxyl groups without the proper catalyst and curing condition.

The general definition of a catalyst is a substance that increases the speed of reaction by lowering the activation energy of the system, and is not consumed during course of reaction. A catalyst, however, cannot initiate a reaction that will not occur by itself (Gagliardi, 1956). The amount of catalyst used in a reaction depends on the reactivity of the reactants and the time and temperature of curing (Cooke & Weigmann, 1982a). Any reactants having a low rate of reaction with cellulose require the use of a stronger catalyst and/or higher temperature and longer time for curing than do those reactants having a high reactivity. The presence of adequate catalysts in the application of durable press chemical finishes is one factor affecting the performance of fabrics (i.e., wrinkle resistance, shrinkage resistance, smoothness, durability, and whiteness).

Based on the previous rationale, reactants used for durable press finishes do not have their reactivity at the same level. Some reactants have low reactivity, which means they can react with cellulose at a low rate of reaction. Catalyst types, the amount of catalyst, or the temperature and time of curing are factors that can be varied to assist them in improving their reactivity. Conversely, reactants that have a high reactivity have less need for a strong catalyst and/or high temperature and long time curing than do low reactivity reactants. Temperature and time of curing not only affect the rate of reaction but also affect yellowing of cellulosic fabrics. Excessive temperature and time of curing may scorch cellulosic fabrics and cause them to yellow. A temperature of 180 °C is normally used as the curing temperature for formaldehyde-free durable

press agents, such as BTCA, TCA, and CA. This temperature provides good results in durable press performance compared with the results achieved at other temperatures with otherwise the same conditions of treating. Therefore, the constant curing temperature of 180 °C was used in this study. Only the time of curing was varied to examine the effect on the mechanical and durable press properties and whiteness of treated cotton fabrics. A good balance among the reactants in BTCA/IA/AA combinations, the amount of catalyst, and the curing condition can provide good results in the mechanical and durable press properties and whiteness of finished cotton fabrics. Three hypotheses based on the explained rationale are the following.

H1: The ability of each reactant in BTCA/IA/AA combinations will be dependent on each other in BTCA/IA/AA finish formulation, amount of catalyst, and curing time to affect the mechanical and durable press properties and whiteness of finished cotton fabrics differently.

H2: The ability of mole ratio of monomers to catalyst will be dependent on BTCA/IA/AA finish formulations and curing time to affect the mechanical and durable press properties and whiteness of finished cotton fabrics differently.

H3: The ability of curing time will be dependent on BTCA/IA/AA finish formulations and mole ratio of monomers to catalyst to affect the mechanical and durable press properties and whiteness of finished cotton fabrics differently.

Objective

This research sought to determine whether the mechanical, durable press, and whiteness properties of cotton fabric treated by the polymerization-crosslinking of BTCA/IA/AA with pad-dry-cure differ from those properties of cotton fabric treated with conventional DMDHEU or with BTCA-based finishes.

Rationale for the associated hypotheses. Polycarboxylic agents will normally form ester bonds with cellulose, while N-methylol agents (DMDHEU) will form ether bonds with cellulose. The BTCA/IA/AA combinations under polymerization-crosslinking with pad-dry-cure may form a polymer that contains enough carboxylic groups to provide ester crosslinking with cellulose as effectively as BTCA does, or as effectively as DMDHEU does in providing ether-crosslinking with cellulose. Moreover, if the polymer formed by the BTCA/IA/AA reaction has a long enough chain to provide flexible crosslinks in cotton fibers, cotton fabrics treated with such a polymer may perform well both in wrinkle resistance and strength retention. Two hypotheses based on this rationale are the following.

H4: Polymerization-crosslinking treatment using BTCA/IA/AA combinations with pad-dry-cure and the BTCA treatment with pad-dry-cure will affect the mechanical and durable press properties and whiteness of treated cotton fabric differently.

H5: Polymerization-crosslinking treatment using BTCA/IA/AA combinations with pad-dry-cure and the conventional DMDHEU treatment with pad-dry-cure will affect the mechanical and durable press properties and whiteness of treated cotton fabric differently.

Assumptions

The following assumptions were made for conducting experiments in this study:

1. It was assumed that all instruments used for this study were accurate and precise.
2. It was assumed that equipment and operator errors were of a random nature and had no significant effect on the testing results.
3. It was assumed that all the reagent-grade chemicals used for this study were consistent in their properties from lot to lot.
4. It was assumed that variations in weight, thickness, and physical properties of the 3/1 twill cotton woven fabric, as received for use in this research, were random.

Limitations

The following limitations were established for conducting experiments in this study:

1. Only 100% cotton, 3/1 twill woven fabric was used in this study.
2. The durable press finishes were applied only on white fabric that had been scoured, bleached, and mercerized.

CHAPTER IV
Research Procedure

This chapter includes the wrinkle recovery angle and breaking strength results for the fabric finished with various combinations of carboxylic derivatives as were examined in the preliminary analysis, criteria used to select any combination of carboxylic derivatives expected to be used as an effective nonformaldehyde DP finishing agent, variables studied in the selected PCA combinations, measured properties of the fabric finished with the selected PCA combinations, and the experimental procedure and data analysis in the main study using BTCA/IA/AA combinations selected as a result of the preliminary analysis.

Preliminary Analysis of Candidate Reactant Combinations and of Finishing Conditions

Experimental Procedure in the Preliminary Analysis

A preliminary analysis provided the information on how the BTCA/IA/AA combination was selected to study in detail as indicated in previous chapters of this dissertation. The preliminary analysis was conducted to test various possible combinations of unsaturated carboxylic acids and of saturated polycarboxylic acids. Each reactant combination was applied, under the same treatment conditions, to specimens of the same 3/1 twill woven cotton fabric by a polymerization-crosslinking process with pad-dry-cure. This preliminary work included measurement of wrinkle recovery and breaking strength of the finished fabric specimens. Yellowing of the specimens was a third factor considered; the researcher made observations of the yellowing and did not take exact measurements. Results on those three factors were used to narrow the reactant combinations for further study, to analyze different concentrations of the reactant combinations, and to decide whether to use potassium persulfate or hydrogen peroxide as the initiator in the main part of the research. High wrinkle recovery angle, high breaking strength retention, and low degree of yellowing of the finished specimens were the criteria used for selecting, for further study, reactant combinations from among those analyzed in the preliminary work. In addition, results on the three factors were compared with the same factors on fabric specimens finished with DMDHEU or BTCA only. Results on the durable press properties of the fabric finished with DMDHEU or BTCA are the standard that this study attempts to meet.

Yang and Wang (1996) used FT-IR to study the esterification of cotton fabric by seventeen aliphatic and aromatic polycarboxylic acids. The results of ester carbonyl band absorbance for BTCA, TCA, CA, succinic acid (SA), maleic acid (MA), and itaconic acid (IA) were 0.569, 0.562, 0.506, 0.520, 0.551 and 0.493 cm^{-1}, respectively. Those values of ester carbonyl band absorbance showed the ability of each acid's first carboxyl group to form an anhydride intermediate and thus esterify cellulosic fibers. These values showed the relative abilities to form the intermediate: the higher the value, the better the ability of an

acid's first carboxyl group to form the intermediate. The first carboxyl group of each of the above carboxylic derivatives had good ability to form the anhydride intermediate. The second carboxyl group of any of the bifunctional carboxylic acids (MA, IA, and SA), however, was not able to form an anhydride intermediate nor to esterify cellulose as effectively as its first carboxyl group, so bifunctional carboxylic acids were less effective crosslinking agents for cellulose than were BTCA, TCA, or CA. Bifunctional carboxylic acids, on their own, were not effective enough to be good crosslinking agents, but combinations of them or with other polycarboxylic acids, used in the polymerization-crosslinking process with pad-dry-cure, may provide good results in durable press properties.

The polymerization-crosslinking process is a treatment involving two components, polymer builder and crosslinker, and is performed to balance good mechanical properties and good durable press performance of finished fabrics. In various experiments in the preliminary work, MA, IA, or a mixture of them in a 1:1 mole ratio was used as a polymer builder, and BTCA, CA, or SA was used as a crosslinker. TCA was excluded in the preliminary work because it is much more expensive than BTCA. Sodium hypophosphite monohydrate was used as a catalyst. Potassium persulfate or hydrogen peroxide was used as an initiator. The finish formulation used in most of the preliminary work contained 2% crosslinker, 6% polymer builder, 1% polyolefin, 0.2% Triton X-100 as a wetting agent, 1:1.25 mole ratio of monomers to sodium hypophosphite monohydrate, and 1.5% on weight of monomers (% o.w.m.) of initiator. In the mixture of 1:1 mole ratio of MA to IA used as a polymer builder, 2.80% MA and 3.20% IA were used to achieve the 1:1 mole ratio. The adjustment in other finish formulations was as specified in later pages in the notes in Tables 5, 6, and 7. The mixing sequence to prepare the finish solution started with a crosslinker, then polymer builder, polyolefin emulsion, Triton X-100, sodium hypophosphite, and the initiator. After adding the initiator, the solution was mixed for 5 min.

An 11x17 in. piece of cotton fabric was immersed in an aqueous finishing solution for 10 min., followed by passing the fabric through squeeze rolls to give a wet pick-up of 95-110% on weight of fabric (% o.w.f.). Then the fabric was mounted on a frame and predried at 100 °C for 10 min. in an oven. After the 10 min. in the oven, the mounted fabric was removed. After raising the oven temperature to 180 °C, the fabric was returned to the oven for curing at 180 °C for 2 min. The finished fabric was later removed from the frame and rinsed in running hot tap water for 10 min. and re-dried at 100 °C for 5 min. The DMDHEU and BTCA treatments, for comparison with the polymerization-crosslinking, were as follows. The DMDHEU treatment was with 12% DMDHEU, 1.5% magnesium chloride hexahydrate, 1% polyolefin emulsion (PE), and 0.2% Triton X-100, followed by predrying at 100 °C for 5 min. and curing at 160 °C for 3 min. The BTCA treatment was with 6.3% BTCA, 6.5% sodium hypophosphite monohydrate, 1% PE, and 0.2% Triton X-100, followed by predrying at 85 °C for 5 min. and curing at 180 °C for 90 sec.

The wrinkle recovery angle and breaking strength were measured on untreated and treated specimens of the same cotton 3/1 twill woven fabric. The treatments included DMDHEU, BTCA and various polymerization-crosslinking

formulations as described above. The application of the several finish formulations on 11x17 in. pieces of the 3/1 cotton twill fabric yielded one 11x17 in. sample for each finish formulation. Each of those samples was cut into the number of specimens of the dimensions indicated in the standard methods used to measure wrinkle recovery angle and breaking strength. The method used for wrinkle recovery angle was AATCC-Method 66-1989, which requires twelve specimens for the measurement on one fabric; the twelve specimens include three in each of four directions (warp face to face, warp back to back, weft face to face, and weft back to back). The method used for breaking strength was ASTM D5035-95, which requires thirteen specimens per fabric: five specimens in the warp direction and eight specimens in the weft direction. Figure 1 shows the pattern used for cutting the specimens from each 11x17 in. fabric piece, as well as the dimensions of the specimens. The specimen dimensions shown in Figure 1 are one-third the size of the actual dimensions.

Table 2 reports results obtained in the preliminary analysis. The table contains the averages of the measured values for wrinkle recovery angle and breaking strength retention of the fabric untreated, treated with DMDHEU, and treated with ten different finish formulations of PCA using potassium persulfate as the initiator. Experiment #1 in Table 2 refers to the untreated fabric, and Experiments #2 and #3 to the DMDHEU treated fabric and the BTCA treated fabric, respectively. Three different systems of polymer builder in the polymerization-crosslinking were studied, those being MA, IA, and MA/IA mixture, as well as three different systems of crosslinkers, BTCA, CA, and SA. The use of these systems built up nine different combinations of treatments with the potassium persulfate initiator. Wrinkle recovery and breaking strength retention results for fabrics finished with the nine different solutions for which potassium persulfate was the initiator are shown in Experiments #4 to #12 in Table 2.

Table 2 reports mean values for wrinkle recovery and breaking strength retention in a manner typical in textile research publications. In Table 2, each average wrinkle recovery angle is the sum of the average wrinkle recovery angles in the two directions, warp and weft, of all specimens in the relevant finished sample. The sum of the average wrinkle recovery angles in both directions was rounded up to a whole number. The percentage breaking strength retention is reported, instead of the breaking strength, in Table 2 because the breaking strength retention better indicates the degree of strength left in the finished fabric. The breaking strength in kilograms is needed, however, for statistical analyses, such as t-Test and regression, and is used in statistical data analysis later in the main part of the study. The breaking strength retention for both the warp and weft directions is reported in Table 2.

The percentage breaking strength retention was calculated by first averaging the breaking strength for all specimens taken from each direction divided by the average breaking strength of untreated fabric in the same direction, and then multiplying by 100. The breaking strength retention results were rounded up to whole numbers. The average breaking strength of untreated fabric was considered to be 100% retention for each direction. It is seen in Table

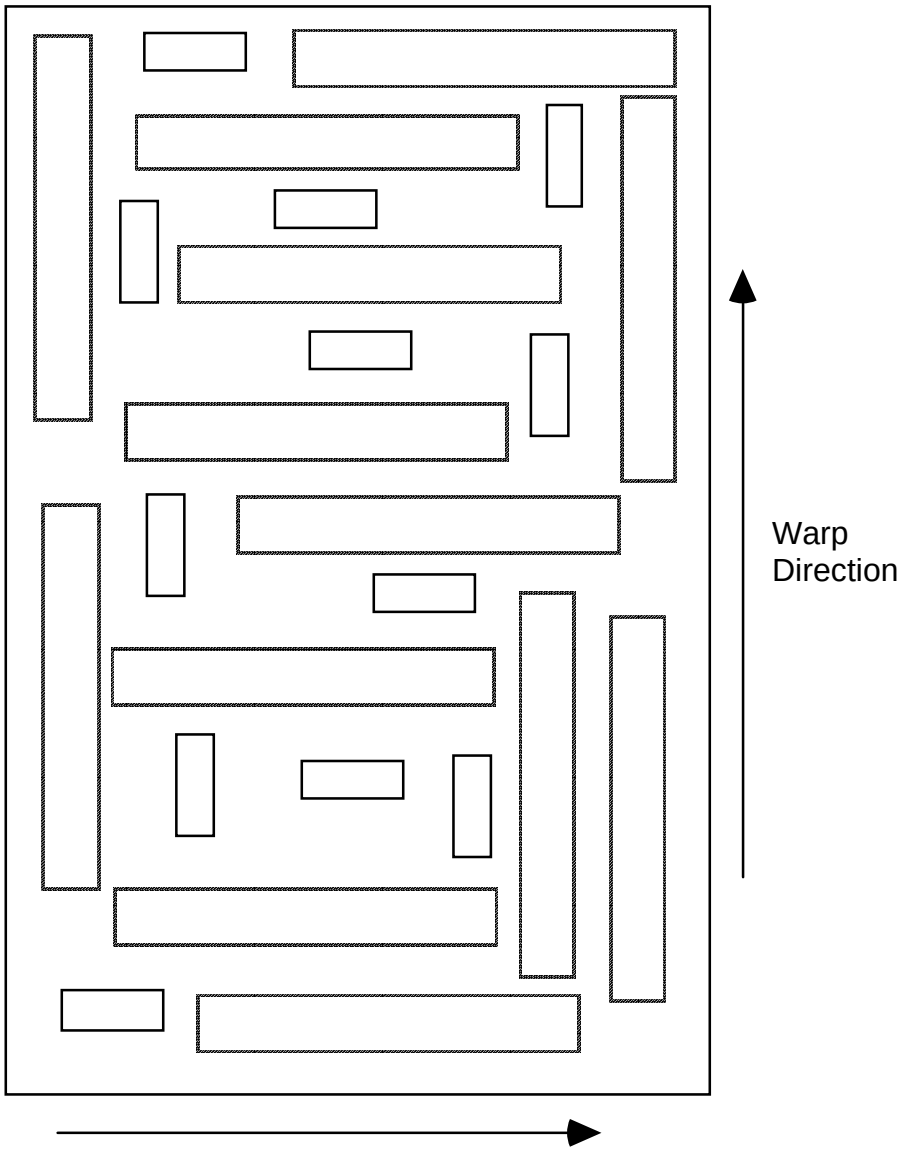


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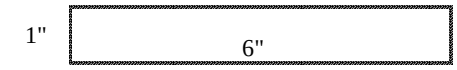
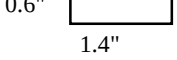
Figure 1. Cutting pattern for specimens to measure breaking strength and wrinkle recovery angle in the preliminary analysis.

Note. Specimen Dimensions



Breaking Strength



Wrinkle Recovery Angle

Table 2 Wrinkle recovery angle (WRA) and breaking strength retention of untreated fabric and of the fabric finished with DMDHEU, BTCA, or the nine different solutions of PCA combinations with potassium persulfate initiator

Experiment	Wrinkle recovery angle, average (Warp + Weft)	Breaking strength retention average Warp	Weft
1. Untreated	146°	100%	100%
2. [a]12%DMDHEU	288°	61%	53%
3. [b]6.3%BTCA	268°	64%	56%
4. [c]2%BTCA/6%MA	260°	66%	55%
5. [c]2%BTCA/6%IA	258°	70%	62%
6. [c]2%BTCA/2.8%MA/3.2%IA	265°	70%	60%
7. [c]2%CA/6%MA	248°	67%	60%
8. [c]2%CA/6%IA	245°	69%	66%
9. [c]2%CA/2.8%MA/3.2%IA	249°	68%	65%
10. [c]2%SA/6%MA	229°	72%	66%
11. [c]2%SA/6%IA	227°	71%	70%
12. [c]2%SA/2.8%MA/3.2%IA	216°	76%	70%

Note. [a]12% DMDHEU, 1.5% magnesium hexahydrate, 1% polyolefin emulsion softener, and 0.2% Triton X-100, followed by predrying at 100 °C for 5 min. and curing at 160 °C for 3 min.

[b]6.3% BTCA, 6.5% sodium hypophosphite monohydrate, 1% polyolefin emulsion softener, and 0.2% Triton X-100, followed by predrying at 85 °C for 5 min. and curing at 180 °C for 90 sec.

[c]2% crosslinker, 6% polymer builder, 1% polyolefin emulsion softener, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min.

2 that, of the results for Experiments # 4 to #12, the PCA combinations containing succinic acid (SA) provided wrinkle recovery angles that were the lowest and that were considerably lower than any other of the finish formulations provided. Thus, SA combinations were eliminated from further study. The raw data on wrinkle recovery angle and breaking strength of the specimens finished with each of the above finish formulations in Table 2 are shown in Appendices A and B, respectively (the averages and standard deviations of those results are also included in Appendices A and B).

Initiator Selection for the PCA Combinations

The next step of the preliminary analysis was to determine whether potassium persulfate or hydrogen peroxide had the better ability to initiate the polymerization of the unsaturated carboxylic acids. In addition to the experiments performed previously with potassium persulfate, hydrogen peroxide was used as the initiator with the six different PCA combinations remaining after eliminating the SA combinations from further consideration. The results on wrinkle recovery angle and breaking strength retention of the fabric finished with those six different PCA solutions, using hydrogen peroxide as the initiator, are shown in Table 3.

Comparing results in Tables 2 and 3, the fabric finished with CA combinations using hydrogen peroxide had lower wrinkle recovery angles (226° to 230°) than the fabric finished with the same CA combinations using potassium persulfate (245° to 249°). The fabric finished with CA combinations under either initiator had similar results in breaking strength retention. For only the CA/IA combination, the result in breaking strength retention was better when using hydrogen peroxide than when using potassium persulfate; that better result was in the warp direction. The fabric finished with BTCA combinations using hydrogen peroxide had smaller wrinkle recovery angles overall than did the fabric finished with the same BTCA combinations using potassium persulfate. The small differences in the breaking strength retention percentages of the fabric finished with different BTCA combinations made it impossible to say whether potassium persulfate or hydrogen peroxide was the better initiator.

In general, PCA combinations using potassium persulfate provided better results in wrinkle recovery angle than did those using hydrogen peroxide, and the PCA combinations using the two different initiators provided similar results in breaking strength retention. Therefore, potassium persulfate was chosen for use as the initiator for further study. The raw data on wrinkle recovery angle and breaking strength of the specimens finished with each of the above finish formulations in Table 3 are shown in Appendices C and D, respectively (the averages and standard deviations of those results are also included in Appendices C and D).

Effect of Nitrogen Atmosphere on Potassium Persulfate Initiator

It has been recommended that the polymerization-crosslinking process involving a reducing agent such as potassium persulfate should be done in a nitrogen atmosphere to prevent the inhibition of polymerization by the dissolution of the oxygen in water and/or in air. Only BTCA combinations with potassium persulfate were studied under N_2 atmosphere because BTCA combinations

Table 3 Wrinkle recovery angle (WRA) and breaking strength retention of fabric finished with six different PCA combinations using hydrogen peroxide initiator

Experiment	Wrinkle recovery angle, average (Warp + Weft)	Breaking strength retention average Warp	Weft
1. [a]2%BTCA/6%MA	266°	63%	60%
2. [a]2%BTCA/6%IA	245°	68%	65%
3. [a]2%BTCA/2.8%MA/3.2%IA	252°	66%	63%
4. [a]2%CA/6%MA	226°	70%	67%
5. [a]2%CA/6%IA	226°	75%	66%
6. [a]2%CA/2.8%MA/3.2%IA	230°	68%	66%

Note. [a]2% crosslinker, 6% polymer builder, 1% polyolefin emulsion softener, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator hydrogen peroxide, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min.

provided the best wrinkle recovery angle. The results on wrinkle recovery angle and breaking strength retention of the fabric finished with BTCA combinations under nitrogen atmosphere are shown in Experiments #1 to #3 in Table 4. The wrinkle recovery angles of fabric finished under nitrogen atmosphere were expected to be higher than the fabric finished under ambient conditions with potassium persulfate initiator in Experiments #4 to #6 in Table 2, but they were not.

The unexpected lack of difference might come from a few steps done differently under N_2 atmosphere, because those steps were tried to remove the oxygen in the water and in the finishing solutions. First, the distilled water, which was used instead of tap water, was boiled for 10 min. Second, the bubble of nitrogen was run through the finishing solution for 5 min. before the fabric was immersed in the solution.

According to the unexpected wrinkle recovery angle results of the fabric finished with BTCA combinations under nitrogen atmosphere, the system of BTCA combinations without initiator was also studied to see how the initiator affected the wrinkle recovery angle results of the fabric finished with BTCA combinations with initiator. The wrinkle recovery angle and breaking strength retention results of the fabric finished with BTCA combinations without initiator are shown in Table 4 as Experiments #4 to #6. The raw data on wrinkle recovery angle and breaking strength of the specimens finished with each of the above finish formulations in Table 4 are shown in Appendices E and F, respectively (the averages and standard deviations of those results are also included in Appendices E and F). The wrinkle recovery angle results in Table 4 are for fabric finished with the two different BTCA systems; one system with initiator under nitrogen atmosphere and the other without initiator under ambient condition had similar results, but the wrinkle recovery angle results from those two systems were less good than those from BTCA combinations with potassium persulfate under ambient condition in Experiments #4 to #6 in Table 2. Some breaking strength retention results for the fabric finished with the BTCA combinations under atmospheric nitrogen with potassium persulfate initiator were better than for the fabric finished with the same BTCA combinations under ambient conditions with or without initiator, but some were not. It was difficult, therefore, to determine which system among the three BTCA systems resulted in the best breaking strength retention; this led to using the results on wrinkle recovery angle as the main criterion for selecting which BTCA combinations to study further.

According to the preliminary results so far, the BTCA combinations with potassium persulfate under ambient conditions provided the best wrinkle recovery angles compared with the wrinkle recovery angles resulting from the other PCA combinations. Finishing solutions of BTCA combinations applied under ambient conditions with potassium persulfate as the initiator were selected for further study in the preliminary analysis.

Problems with BTCA Combinations

Some disadvantages of certain BTCA combinations were observed in the experimentation. The BTCA/MA combination showed a tendency to precipitate

Table 4 Wrinkle recovery angle and breaking strength retention of fabric finished with BTCA combinations under nitrogen atmosphere and those with the same combinations without initiator under ambient condition

Experiment	Wrinkle recovery angle, average (Warp + Weft)	Breaking strength retention average Warp	Weft
1. [a]2%BTCA/6%MA	247°	62%	61%
2. [a]2%BTCA/6%IA	242°	71%	65%
3. [a]2%BTCA/2.8%MA/3.2%IA	251°	68%	65%
4. [b]2%BTCA/6%MA	249°	67%	62%
5. [b]2%BTCA/6%IA	240°	75%	67%
6. [b]2%BTCA/2.8%MA/3.2%IA	247°	68%	63%

Note. [a]2% BTCA, 6% polymer builder, 1% polyolefin emulsion softener, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate under atmospheric nitrogen, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min.

[b]2% BTCA, 6% polymer builder, 1% polyolefin emulsion softener, 0.2% Triton X-100, and 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min. (no initiator).

the sparingly soluble monosodium maleate. Welch and Peters (1997) also found this precipitate problem of the BTCA/MA combination. In the preliminary analysis in the present research, adding more water in a finish formulation than normally used or increasing the temperature of the mixing solution to between 40 and 50 °C before fabric immersion prolonged the precipitation. Welch and Peters (1997) suggested that adding 1.5% phosphoric acid and keeping the treating bath warmed to 35 °C during application to the fabric would redissolve the precipitate of monosodium maleate. The BTCA/MA combination was not examined further in the present study because of the great care required to avoid the precipitation problem of monosodium maleate. The one advantage of the BTCA/MA combination was that it did not yellow the finished fabric. All the other studied PCA combinations caused the observed yellowing of the finished fabric. The yellowing was obvious when the fabric specimens were removed from the oven after the curing step. The observed yellowing of the fabric finished with CA combinations was more severe than that of fabric finished with BTCA combinations.

The yellowing of the fabric finished with the BTCA/MA/IA and BTCA/IA combinations typically became apparent after the curing at 180 °C for 2 min. Fung, Wong, and Brotherton (1993) suggested that 1-3% boric acid added to a finishing solution markedly reduces the yellowing resulting from the high temperature treatment of cellulosic textiles. Thus, the BTCA/MA/IA finish formulation containing 0.5% o.w.b. boric acid was studied in the preliminary work. The resulting average wrinkle recovery angle was 239°, and the breaking strength retention was 64% in the warp direction and 62% in the weft. The cotton fabric finished with this solution had less observed yellowing than that finished with the same solution but without boric acid (Experiment #6 in Table 2). The two BTCA/MA/IA systems, with and without boric acid, showed a significant difference at the .05 level in the wrinkle recovery property of the finished fabric ($F(1, 16) = 43.19$, $p = 0.0001$), but no significant difference in breaking strength retention of the finished fabric ($F(1,22) = 0.78$, $p=0.3877$). Although boric acid appears to have affected the wrinkle recovery angles but not the breaking strength retention of the finished fabric in this case, the effect of boric acid on the durable press performance of finished fabric needs more study and was not pursued further in this research. Because the BTCA/IA system without boric acid appeared to cause more observed yellowing of the finished fabric than did the BTCA/MA/IA system without boric acid, the BTCA/IA system might need to have a greater amount of boric acid added to reduce yellowing; adding boric acid in the system might cause the side effect of poor wrinkle recovery in the durable press performance of the finished fabric. Analysis of such effects of boric acid is beyond the scope of this research.

In the preliminary study, not only BTCA/IA combination had the yellowing effect on the finished fabric but also CA combinations. The CA/MA combination had the two problems of the precipitation of monosodium maleate and the yellowing of the finished fabrics. The CA/MA/IA combination tended to cause yellowing also, but it did not cause the precipitate at the concentration that was studied. Precipitation could occur with increasing concentration of the reactants

and the concomitant need for more sodium hypophosphite monohydrate. The CA/IA combination had only the problem of yellowing; therefore, the CA/IA combination was chosen to study the effect on wrinkle recovery angle and breaking strength retention of adding 2% BTCA in a finishing solution. A reason to add BTCA in the CA/IA finishing formulation was because Welch and Peters (1997) found that the addition of BTCA to a CA finishing formulation can improve the durable press performance of the finished fabrics. A reason to add 2% BTCA was because this concentration of BTCA had been used at the beginning of the preliminary analysis. The main part of the research required more pieces of finished fabric for five different measurements, not only for wrinkle recovery angle and breaking strength but also for tear strength, whiteness, and durable press rating. Thus, the effect of reusing the finishing solution needed be studied to determine whether the finishing solution could or could not be reused and, if reused, how many times. The effects of reusing the 2%BTCA/2%CA/6%IA finishing solution system and of increasing the IA concentration were studied. Increasing the IA concentration was studied to determine how increased IA concentration affects wrinkle recovery angle and breaking strength retention of the finished specimens. The results on wrinkle recovery angle and breaking strength retention for the fabric finished with various BTCA/CA/IA combinations are shown in Table 5. The raw data on wrinkle recovery angle and breaking strength of the specimens finished with each of the above finish formulations in Table 5 are shown in Appendices G and H, respectively (the averages and standard deviations of those results are also included in Appendices G and H).

The wrinkle recovery angle and breaking strength retention results for the fabric finished in the first use of the 2%BTCA/2%CA/6%IA combination solution and for the fabric finished in the second use of the same solution are shown in Experiments #1 and #2 in Table 5. Wrinkle recovery angles from those two finished fabric specimens (266.5° and 267°) were not significantly different (F (1, 16) = 0.89, p = 0.3597). The breaking strength retention was not significantly different between the two finished fabric specimens (F (1, 22) = 2.37, p = 0.1378). One could conclude that the treating solution could be reused, but how many times it could be reused needs more study.

The BTCA/CA/IA combination was studied further by increasing the concentration of IA from 6% o.w.b. to 9% o.w.b. to determine the effect on wrinkle recovery angle and breaking strength retention of the finished fabric. The wrinkle recovery angle and breaking strength retention of the fabric finished with the 2%BTCA/2%CA/9%IA solution are shown in Experiment #3 in Table 5. The two different systems of IA concentrations in Experiments #1 and #3 resulted in significant differences in wrinkle recovery angle (F (1, 16) = 39.92, p = 0.0001) and in breaking strength retention (F (1, 22) = 11.53), p=0.0026).

The wrinkle recovery angle and breaking strength retention results in Experiment #1 in Table 5 of the 2%BTCA/2%CA/6%IA combinations were similar to those results of the fabric finished with only the 6.3% BTCA finish formulation; however, the 2%BTCA/2%CA/9%IA treatment provided significantly different results in the wrinkle recovery angle property (F (1, 16) = 5.98, p = 0.0274) compared to those of the 6.3% BTCA reactant. These two treatments also

Table 5 Wrinkle recovery angles and breaking strength retentions of the fabric finished with two different BTCA/CA/IA combinations: study of BTCA addition to CA/IA; solution reusability; and IA concentration

Experiment	Wrinkle recovery angle, average (Warp + Weft)	Breaking strength retention average Warp	Weft
1. [a]2%BTCA/2%CA/6%IA	267°	67%	59%
2. [b]2%BTCA/2%CA/6%IA	267°	66%	56%
3. [c]2%BTCA/2%CA/9%IA	276°	57%	56%

Note. [a]2% BTCA, 2% CA, 6% IA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min. Measurement results were taken from the fabric finished in the first use of the solution.

[b]The same finishing formulation as above, but measurement results were taken from the fabric finished in the second use of the solution.

[c] 2% BTCA, 2% CA, 9% IA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min.

provided significant differences in breaking strength retention results (F (1, 22) = 26.81, p = 0.0001). The fabric finished with the 2% BTCA/2% CA/9% IA did better in wrinkle recovery angle (276°) compared to that (268°) of the fabric finished with BTCA only, but worse in breaking strength retention (57.0% in warp and 55.7% in weft) compared to those with BTCA only (63.6% in warp and 55.6% BS retention in weft).

The disadvantage of the two finish formulations of BTCA/CA/IA combinations was the discoloration of the finished specimens. The discoloration was obvious when the specimens were taken out of the oven after the curing step. The BTCA/CA/IA combination might be usable as an effective durable press finish, but it needs more study to solve the problem of yellowing. The BTCA/CA/IA combinations were not studied further in this research because of the severe yellowing problem on the specimens.

Adding Acrylic Acid as a New Polymer Builder

In addition to MA and IA, acrylic acid (AA) was another unsaturated carboxylic acid assessed as a polymer builder in the preliminary study. The acrylic acid contained the inhibitor to prevent polymerization during shipping. The amount of the initiator and catalyst used in the study were provided sufficiently to overcome the inhibitor, therefore, it was used in the study without removing the inhibitor. AA polymerizes to a longer polymer easier than IA or MA. The wrinkle recovery angle and breaking strength retention of the fabric finished with various solutions containing acrylic acid are shown in Table 6. The initiator used in these finishing solutions was potassium persulfate. The 2%BTCA/6%AA or 2%CA/6%AA combinations provided wrinkle recovery angles of the finished fabric specimens of only 204° and 184°, respectively (Experiments #1 and #2 in Table 6), which are the lowest wrinkle recovery angles of those reported in Table 6 and indeed are the lowest wrinkle recovery angles of finished specimens in the preliminary analysis. Table 6 also reports results obtained for finished specimens when a mixture of 1:1 mole ratio of AA and either MA or IA was used as a polymer builder.

One polymer builder mixture of 1:1 mole ratio was achieved with 3.2%MA, and 1.77% AA, and the other was achieved with 3.2% IA and 1.77% AA. Fabric specimens finished with 2%BTCA/3.2%IA/1.77%AA or 2%BTCA/3.2%MA/1.77%AA (Experiments #3 and #4 in Table 6) did not differ significantly in the results on wrinkle recovery angles (F (1, 16) = 0.88, p = 0.3633) and on breaking strength retention (F (1,22) = 2.45, p = 0.1319). Because IA is cheaper than MA, the BTCA/IA/AA combination was chosen for additional preliminary analysis by varying the mole ratio of IA/AA mixtures and the curing times.

In Experiment #5, noted in Table 6, the concentrations of BTCA/IA/AA remained the same as in Experiment #4 in Table 6, but the curing time was increased from 2 min. to 3 min. The mechanical properties of the finished specimens in Experiments #4 and #5 were compared. The longer curing time did not significantly affect the wrinkle recovery angle of the finished fabric (F (1,16) = 3.23, p = 0.0911), but it significantly affected the breaking strength

Table 6 Wrinkle recovery angle and breaking strength retention of the fabric finished with various finish formulations containing acrylic acid

Experiment	Wrinkle recovery angle, average (Warp + Weft)	Breaking strength retention average Warp	Weft
1. [a]2%BTCA/6%AA	204°	74%	65%
2. [a]2%CA/6%AA	184°	77%	74%
3. [b]2%BTCA/3.2%MA/1.77%AA	234°	64%	62%
4. [b]2%BTCA/3.2%IA/1.77%AA	237°	68%	64%
5 [c]2%BTCA/3.2%IA/1.77%AA	242°	63%	60%
6. [d]2%BTCA/6.4%IA/1.77%AA	256°	63%	60%
7. [e]2%CA/6.4%IA/1.77%AA	241°	67%	60%
8. [f]4%CA/6.4%IA/1.77%AA	258°	63%	57%
9. [g]3%BTCA/6.4%IA/1.77%AA	268°	64%	55%

Note. [a]2% crosslinker (BTCA or CA), 6% AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min.

[b]2% BTCA, 1:1 mole ratio of the mixture of polymer builder, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 2 min.

[c]2% BTCA, 3.2% IA, 1.77% AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 3 min.

[d]2% BTCA, 6.4% IA, 1.77% AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 3 min.

[e]2% CA, 6.4% IA, 1.77% AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 3 min.

[f]4% CA, 6.4% IA, 1.77% AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 3 min.

[g]3% BTCA, 6.4% IA, 1.77% AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 3 min.

retention (F (1,22) = 7.64, p = 0.0113). The longer curing time decreased the breaking strength retention of the fabric under these finishing conditions.

In Experiment #6, noted in Table 6, the same curing time of 3 min. was used as in Experiment #5 in Table 6, but the mole ratio of IA/AA was increased from 1:1 to 2:1 in the BTCA/IA/AA combinations. The 2:1 mole ratio of IA to AA was achieved with 6.4% IA and 1.77% AA. Under the fabric finishing conditions used in Experiments #5 and #6, the 2:1 mole ratio of IA/AA resulted in wrinkle recovery angles of about 10° more than the wrinkle recovery angles that resulted with 1:1 mole ratio of IA to AA (F (1, 16) = 18.59, p = 0.0005), while the breaking strength retention of the fabric specimens was not significantly different (F (1, 22) = 0.02, p = 0.8906). The BTCA/IA/AA combinations did not cause as much observed yellowing on the finished fabric as the BTCA/IA combinations did, as mentioned before on page 56. The double bonds left in the finish formulation after the reaction of polymer builder systems were expected to cause yellowing on the finished fabric. Adding AA in the system may have improved the polymerization process of IA so the finish formulation had fewer double bonds.

Because AA appeared to reduce the yellowing problem in combinations containing BTCA and IA, it was suspected that AA might do as well in combinations containing CA and AA. This potential effect was investigated in Experiments #7 and #8, noted in Table 6, in which finish formulations of 2% or 4% CA and 2:1 mole ratio of IA to AA were used. The concentration of 2% CA in the CA/IA/AA combination did not cause obviously observed yellowing on the finished fabric, but the concentration of 4% CA in the CA/IA/AA combination tended to cause quite obvious yellowing on the finished fabric when the yellowing was compared with that obtained from 2% CA in the CA/IA/AA combination. Measurement results on mechanical properties of fabric finished with the CA/IA/AA combinations in Experiments #7 and #8 are shown in Table 6. The finish with 4% CA and 2:1 mole ratio of IA to AA gave quite good wrinkle recovery angles of 258° on average, as compared to the 241° with 2% CA; the breaking strength retention was comparable to that with the other finishing formulations of BTCA/IA/AA. The CA/IA/AA combination did not include an expensive chemical like BTCA, so this may be a PCA combination that can serve as a cheap durable press finish; however, because it had the disadvantage of the yellowing problem, as observed in one of the experiments, CA/IA/AA was not selected to study further in this research.

Increasing BTCA from 2% to 3% in the BTCA/6.4% IA/1.77% AA treatment was studied to observe how much the additional 1% of BTCA could affect the wrinkle recovery angles and breaking strength retention of the finished fabric. The 3%BTCA/6.4%IA/1.77%AA solution (Experiment #9 in Table 6) provided an average wrinkle recovery angle of 268° and breaking strength retention of 64% and 55% in the warp and weft directions, respectively. The wrinkle recovery angle and breaking strength retention results of the fabric finished with this combination with 3% BTCA were comparable to the results of the fabric treated with DMDHEU or BTCA reactants alone in Experiments #2 and #3 in Table 2. Comparing the wrinkle recovery angle and breaking strength retention of fabric finished with 3% BTCA and 2:1 mole ratio of IA/AA with those

properties of fabric finished with BTCA only, the wrinkle recovery angles were not significantly different (F (1,16) = 0.1, p = 0.7544), nor was the breaking strength retention (F (1,22) = 1.34, p = 0.2589). Comparing those properties of fabric finished with that same BTCA/IA/AA combination with those of fabric finished with DMDHEU, the wrinkle recovery angles were significantly different (F (1,16) = 122.00, p = 0.0001) and the breaking strength retention was not (F (1, 22) = 2.59, p = 0.1220). The fabric finished with the 3%BTCA/6.4%IA/1.77%AA solution had the same wrinkle recovery angles as that treated with 6.3% BTCA, but lower wrinkle recovery angles than that treated with 12% DMDHEU. The fabric finished with 3%BTCA/6.4%IA/1.77%AA had the same breaking strength retention as that finished with DMDHEU or with BTCA. No precipitation, nor fabric yellowing, was observed with this or any other BTCA/IA/AA combinations; therefore, the BTCA/IA/AA combinations were chosen to study further in the research. The raw data on wrinkle recovery angle and breaking strength results of each of the fabric specimens finished with the various above treatments containing acrylic acid are shown in Appendices I and J, respectively (the averages and standard deviations of those results are also included in Appendices I and J).

Augmenting the study of one reuse of a solution of a BTCA combination, discussed in relation to Experiments #1 and #2 in Table 5, the 3%BTCA/6.4%IA/1.77%AA finishing solution was studied to examine the effect of reusing the solution six times. Six pieces of 11x17 in. 3/1 cotton twill fabric were finished one by one with the same solution. A piece of the fabric was soaked in the finishing solution for 10 min., and then it was removed from the solution and run through the other steps of the finishing process. The second fabric piece was soaked next in the same solution and removed after another 10 min. The remaining pieces of fabric were finished in the same way with the reused solution. These six pieces of fabric were enough to provide all specimens needed for the five tested properties, which will be discussed in the topic of experimental design. The results on wrinkle recovery angle and breaking strength retention for each of the six different fabric specimens finished with the 3%BTCA/6.4%IA/1.77%AA solution are shown in Table 7.

There were no significant differences in the wrinkle recovery angles (F (5, 48) = 0.86, p = 0.5126) nor in the breaking strength retention (F (5, 66) = 1.50, p=0.2006) of specimens from the six different fabric pieces. Reusing the BTCA/IA/AA solution six times did not affect either tested property of the finished fabric specimens. The same conclusion was reached in reusing the solution of BTCA/CA/IA twice (Experiments #1 and #2 in Table 5). Therefore, each finish formulation of BTCA/IA/AA combinations was reapplied on six different pieces of fabric in the main study that followed. The raw data on wrinkle recovery angle and breaking strength results of the six fabric pieces finished with the same finish formulations of the 3%BTCA/6.4%IA/1.77%AA combination are shown in Appendices K and L, respectively (the averages and standard deviations of those results are also included in Appendices K and L).

Table 7 Wrinkle recovery angle and breaking strength retention of the six finished fabric pieces finished with the same solution of 3%BTCA/6.4%IA/1.77%AA combination

3%BTCA/6.4%IA/1.77%AA Solution	Wrinkle recovery angle, average (Warp + Weft)	Breaking strength retention average Warp	Weft
1. 1st fabric piece	266°	64%	58%
2. 2nd fabric piece	263°	62%	61%
3. 3rd fabric piece	264°	63%	59%
4. 4th fabric piece	262°	66%	60%
5. 5th fabric piece	266°	66%	60%
6. 6th fabric piece	266°	66%	61%

Note. Processing conditions included 3% BTCA, 6.4% IA, 1.77% AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1.25 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate, followed by predrying at 100 °C for 10 min. and curing at 180 °C for 3 min.

Summary of the Experimental Results in the Preliminary Analysis

The first step of the preliminary analysis was to finish the cotton fabric with nine different PCA combinations of the three different crosslinkers, BTCA, CA, and SA, and three different polymer builders, MA, IA, and MA/IA mixture, with potassium persulfate initiator. The results on wrinkle recovery angle and breaking strength retention in Table 2 indicate that the PCA combinations containing SA as a crosslinker provided the lowest wrinkle recovery angles compared with those with the other finish formulations analyzed. Thus, the SA combinations were eliminated from further study.

The second step of the preliminary analysis was to determine whether potassium persulfate or hydrogen peroxide had the better ability to initiate the polymerization of the polymer builders in the six different PCA combinations remaining after eliminating the SA combinations in the first step. The fabric finished with either BTCA combinations or CA combinations using potassium persulfate had larger wrinkle recovery angles overall than did the fabric finished with the same PCA combinations using hydrogen peroxide (based on wrinkle recovery angle and breaking strength retention results in Tables 2 and 3). The breaking strength retention of the finished fabric specimens was comparable with both PCA combinations systems using the two different initiators. Therefore, BTCA combinations with potassium persulfate initiator were chosen for further study due to the better results in the wrinkle recovery angles of the finished fabric specimens. Potassium persulfate is a reducing agent. The oxygen in air and water could inhibit the ability of potassium persulfate to initiate the polymerization. The pad-dry-cure process with BTCA combinations using potassium persulfate initiator under atmospheric nitrogen and with BTCA combinations without initiator under ambient condition were studied to compare with the BTCA combinations using potassium persulfate under ambient condition. It was difficult to determine which system among the three systems of BTCA combinations resulted in the best breaking strength retention of the finished fabric specimens. Thus, the wrinkle recovery angle results were used to select the best system of BTCA combinations to study further. According to the preliminary results in Tables 2 and 4, the system of BTCA combinations with potassium persulfate initiator under ambient condition provided the best wrinkle recovery angles compared with those other two systems; therefore, it was selected for further study in the preliminary work.

Some disadvantages of certain BTCA combinations were found in the study. The BTCA/MA combination showed a tendency to precipitate the sparingly soluble monosodium maleate. Welch and Peter (1997) suggested that adding 1.5% phosphoric acid and keeping the treating bath warmed to 35 °C during application on the fabric would redissolve the precipitate of monosodium maleate. Although their suggestion might have solved the precipitate problem of the BTCA/MA combination, this combination was not examined further because of the great care required to avoid the precipitation of monosodium maleate. The BTCA/MA/IA and BTCA/IA combinations showed a tendency to yellow the finished fabric specimens after the curing at 180 °C for 2 min. In the preliminary study, adding boric acid reduced the yellowing, but it deteriorated the durable

press performance of the finished fabric. Because the BTCA combinations, which were studied up to this point, had caused problems in either yellowing or precipitation of monosodium maleate, acrylic acid (AA) was tried as a new polymer builder for the PCA combinations.

Acrylic acid was added in various PCA combinations, as shown in Table 6. It was concluded that the 3%BTCA/6.4%IA/1.77%AA combination did not provide obvious yellowing on the finished fabric specimens, and it provided good results on the wrinkle recovery angle and breaking strength retention of the finished fabric, those results being comparable to the results of fabric finished with either BTCA or DMDHEU reactants. It was decided to change the relative concentrations of the reactants of the BTCA/IA/AA combinations in further study for possible improvement in the breaking strength of the finished fabric. The other conclusions that were found in the preliminary study were: the finish solution of BTCA/IA/AA could be reused for six times without affecting the wrinkle recovery angle and breaking strength retention of the finished fabric; the different directions, warp or weft, of the finished fabric specimens differed in the wrinkle recovery angle and breaking strength retention properties; and the amount of 1.5% o.w.m. of potassium persulfate initiator, the drying conditions of 100 °C for 10 min., and the curing temperature of 180 °C were suitable for driving the polymerization reaction of PCA combinations to occur in a short time and to impart good wrinkle recovery.

This chapter continues by describing the experimental procedure and the evaluations of the mechanical properties of the finished cotton fabric specimens and the experimental design of the main study using BTCA/IA/AA combinations.

Experimental Procedure of the Main Study

This section describes the characteristics of the fabric and chemical agents used in durable press finishing with BTCA/IA/AA combinations, the fabric finishing method, and the procedure for measuring the mechanical and durable press properties and the whiteness of the fabric specimens.

Material and Chemical Agents Used in Durable Press Finishing

The fabric used in the research was a 100% cotton 3/1 twill woven fabric that had been scoured, bleached, and mercerized by Cotton Incorporated which supplied the fabric. The fabric count was 104x48 per square inch and the fabric weight was 7.61 ounces per square yard as measured by the researcher. The reactants used in the PCA finish formulations were laboratory-grade, at 99% purity, itaconic acid, acrylic acid, and 1,2,3,4-butanetetracarboxylic acid that were purchased from Aldrich Chemical Company, Inc. The acrylic acid, as purchased and used in the research, contained 200 ppm. hydroquinone monomethyl ether as an inhibitor to prevent dimerization. Dimethyloldihydroxyethylene urea (DMDHEU) (Freerez™ 900), a commercial product in the form of a 44% solid solution supplied by Freedom Textile Chemicals Co., was also used in fabric finishing. Laboratory-grade potassium persulfate initiator and sodium hypophosphite monohydrate catalyst, for finishing with PCA formulations, were purchased from Sigma Chemical Co. Laboratory-grade magnesium chloride hexahydrate catalyst, for finishing with DMDHEU,

was purchased from Fisher Chemical. The nonionic wetting agent Triton X-100 was purchased from Rohm and Haas Co. Polyolefin emulsion softener (PAT-SOFT-PHD) was supplied by Yorkshire Pat-Chem.

Method for Fabric Finishing

All the PCA fabric finishing was conducted with polymerization-crosslinking (PC) used with the pad-dry-cure technique. The size of each fabric piece that was finished was 11x17 in. The pan that was used to contain the finishing solutions was a 11.5x17.75 in. non-sticky pan. The cotton fabric was immersed for 10 min. in the aqueous treating solution containing a mixture of IA and AA polymer builders, BTCA crosslinker, polyolefin emulsion softener, Triton X-100, sodium hypophosphite monohydrate, potassium persulfate, and water. Each of the PCA finish formulations is listed in Table 8. The amount of water used in each finish formulation was varied among treatments. The total solution used in this study was 650 ml. The amount of water used in each treatment was the total solution (650 ml) minus the total liquid form of the chemical reactants. The chemicals used in liquid form in this study were DMDHEU, AA, Triton X-100, and polyolefin emulsion. The amount of water was added by judgment to dissolve or to dilute the chemical reactants. After this step, each solution form of chemical reactants was mixed in a 1000-ml. beaker for 5 min., and then the mixed solution was poured into the non-sticky pan. A fabric piece was then immersed into the solution for 10 min. After removal from the immersion bath, the fabric was padded through squeeze rollers to give a wet pick-up of 95-110% o.w.f. The wet pick-up was calculated by taking the weight of an immersed fabric piece minus the weight of a dried fabric piece and multiplied by 100.

The next step was to mount the fabric on a frame and predry it at 100 °C for 10 min. in a Model 625 convection oven (Precision Scientific, Winchester, VA). The mounted fabric was removed from the oven after the 10 min. at 100 °C. The oven temperature was raised to 180 °C, and then the mounted fabric was returned to the oven for curing at 180 °C for either 90 sec. or 3 min. Upon removal from the curing oven, the finished fabric was dismounted from the frame and rinsed in running hot tap water for 10 min. After rinsing, the finished fabric was remounted on the frame and redried in the oven at 100 °C for 5 min. Mounting the fabric on the frame served to simulate the mechanical restraint under which fabrics are dried and cured, as rollers pull them at full width through dryers, in the textile finishing industry. The DMDHEU finishing was conducted by following the same procedures as in preparing the BTCA-only and the BTCA/IA/AA combination formulations. The chemical reactants used in the DMDHEU finishing were DMDHEU and magnesium chloride initiator.

Evaluation of the Performance of the Finished Cotton Fabrics

Measurement of mechanical and durable press fabric properties. Breaking strength, tear strength, wrinkle recovery angle, durable press rating, and whiteness were the properties of the finished cotton fabric that were evaluated. Unfinished fabric also was measured for those tested properties for information about the unfinished fabric properties but not for comparison with the properties of the fabric finished with various durable press finish agents. Breaking strength and tear strength are mechanical properties, and wrinkle recovery angle and durable press rating are durable press properties. All fabric

Table 8 Durable press finish formulations of the 32 treatments

Treatment No.	Reactant Concentrations (% o.w.b.)			Mole Ratio of Acid to Catalyst	Curing Time
	BTCA	IA	AA		
1.	2%	6.4%	1.77%	1:1	3 min.
2.	2%	6.4%	3.54%	1:1	3 min.
3.	2%	9.6%	1.77%	1:1	3 min.
4.	2%	9.6%	3.54%	1:1	3 min.
5.	2%	6.4%	1.77%	1:1	90 sec.
6.	2%	6.4%	3.54%	1:1	90 sec.
7.	2%	9.6%	1.77%	1:1	90 sec.
8.	2%	9.6%	3.54%	1:1	90 sec.
9.	3%	6.4%	1.77%	1:1	3 min.
10.	3%	6.4%	3.54%	1:1	3 min.
11.	3%	9.6%	1.77%	1:1	3 min.
12.	3%	9.6%	3.54%	1:1	3 min.
13.	3%	6.4%	1.77%	1:1	90 sec.
14.	3%	6.4%	3.54%	1:1	90 sec.
15.	3%	9.6%	1.77%	1:1	90 sec.
16.	3%	9.6%	3.54%	1:1	90 sec.
17.	2%	6.4%	1.77%	1:0.8	3 min.
18.	2%	6.4%	3.54%	1:0.8	3 min.
19.	2%	9.6%	1.77%	1:0.8	3 min.
20.	2%	9.6%	3.54%	1:0.8	3 min.
21.	2%	6.4%	1.77%	1:0.8	90 sec.
22	2%	6.4%	3.54%	1:0.8	90 sec.
23.	2%	9.6%	1.77%	1:0.8	90 sec.
24.	2%	9.6%	3.54%	1:0.8	90 sec.
25.	3%	6.4%	1.77%	1:0.8	3 min.
26.	3%	6.4%	3.54%	1:0.8	3 min.
27.	3%	9.6%	1.77%	1:0.8	3 min.
28.	3%	9.6%	3.54%	1:0.8	3 min.
29.	3%	6.4%	1.77%	1:0.8	90 sec.
30.	3%	6.4%	3.54%	1:0.8	90 sec.
31.	3%	9.6%	1.77%	1:0.8	90 sec.
32.	3%	9.6%	3.54%	1:0.8	90 sec.

Note. All the durable press finish formulations mentioned above also contained 1% polyethylene emulsion, 0.2% Triton X-100, and 1.5% o.w.m. potassium persulfate initiator, followed by predrying at 100 °C for 10 min.

specimens were conditioned at 20 °C ± 1 °C and 65 ± 2% relative humidity for 24 hours before measuring the properties. The specimens were not laundered before measuring their properties, except the specimens used in the durable press ratings.

Breaking strength was measured on the Instron 1130 Test Instrument (Instron Crop., Canton, MA), following the ASTM D 5035-95 Standard Test Method for Breaking Force and Elongation of Textile Fabrics (Strip Method). The standard test requires five specimens, each 1 in. wide by 6 in. long, in the warp direction and eight specimens, each 1 in. wide by 6 in. long, in the weft direction.

Tear strength was measured on the Elmendrof Tear Tester (Thwing-Albert Instrument Co., Philadelphia, PA), following the ASTM D1424-83 Standard Test Method for Tear Resistance of Woven Fabrics by Falling-Pendulum (Elmendrof) Apparatus. This standard method does not specify a particular number of specimens for testing. Rather, it indicates an equation to calculate the required number of specimens for testing. The number of specimens calculated from the equation depends on reliable estimates of the coefficient of variation of individual observations on similar materials; however, the test method requires no more than ten specimens. A cutting die for cutting specimens to test on the Elmendrof Apparatus provided the basic rectangular test specimen of 3 in. wide by 4 in. long. Eight specimens total, four in the warp direction and four in the weft direction, were the chosen number for the tear strength test of each finished fabric piece because the dimensions of the piece allowed for only eight specimens.

Wrinkle recovery angle was measured on the Wrinkle Recovery Tester (T.J. Edwards, Inc., Boston, MA), following the AATCC Test Method 66-1990 for Wrinkle Recovery Angle. The standard test method requires twelve specimens 0.6 in. wide by 1.4 in. long, six in the long dimension parallel to the warp and six in the long dimension parallel to the weft. Three specimens from each set of six were creased on one side (face side) and three on the other side (back side).

The durable press rating was taken by comparing fabric specimens with the AATCC 3-D Smoothness Appearance Replicas, in accordance with the AATCC Test Method 124-1989 for Appearance of Fabrics after Repeated Home Laundering. The standard method specifies three 15x15 in. fabric specimens cut parallel to the fabric length and width. In this research, the laboratory machine (having two squeeze rollers) that was used to pad the fabric was 12 in. wide, which governed the specimen size available for the finishing. Each fabric piece was cut to 11 in. wide to leave 0.5 in. from each edge of the machine and for fitting in the finishing pan. The length of the fabric piece for finishing was 17 in. The size of the finished fabric specimen was 11X17 in.; therefore, the square specimen for the DP rating testing was only 11x11 in. The durable press rating of each of the three specimens was evaluated after one and five launderings. In addition, the standard method requires three observers to rate the finished fabric specimens to eliminate biased results from only one observer.

Before carrying out the durable press ratings, the specimens cut for this purpose were machine washed in a MAYTAG Model A806 (MAYTAG Company,

Newton, IA). The washing machine was set at regular wash, regular spin, hot wash, warm rinse, and normal water level, which are settings that correspond to the normal or cotton/sturdy washing machine conditions indicated in the AATCC Test Method 124-1989. AATCC standard detergent 124, in the amount of 90 g, was put in the washing machine first, followed by a 4-lb random mixture of dummy cotton-fabric pieces, having several sizes, and six finished specimens cut for the durable press rating. Three specimens from each of two finish applications were washed at the same time. After the washing machine finished the cycle, all pieces of cloth were dried in a MAYTAG Model DE806 dryer. The dryer control dial was set at regular, which corresponded to the cotton/sturdy dryer condition indicated in the standard method. After one laundering prior to evaluation, each finished specimen was hung vertically from two of its corners to avoid distortion, conditioned at 20 °C ± 1 °C and 65 ± 2% relative humidity for 24 hours, and then evaluated as to its durable press rating. Each of these specimens underwent the same washing-drying sequence four more times so that durable press ratings were recorded after one and five of launderings.

Whiteness was measured to examine the possible yellowing effect of the durable press finishing on the white cotton fabric. Whiteness was measured on a Labscan Color Measuring Instrument (Hunter Associates Laboratory, Inc., Reston, VA), following the AATCC Test Method 110-1989 for Whiteness of Textiles. The whiteness index was calculated by the equation shown below:

$$W = Y + 800(x_n - x) + 1700(y_n - y)$$

where W = whiteness index
Y = CIE tristimulus value Y of the sample
x, y = chromaticity co-ordinates of the sample
x_n = 0.3152, the x chromaticity co-ordinate of a perfect diffuser
y_n = 0.3346, the y chromaticity co-ordinate of a perfect diffuser

Note that the values of x_n and y_n given above were specified for the Labscan Instrument used for measuring whiteness; those values differ from those indicated in the AATCC standard method, which are x_n = 0.3101 and y_n = 0.3162. A total of six readings for whiteness was taken on the Labscan from the three specimens. The diameter of a circle for measuring the whiteness was 1.75 in. The finished fabric specimen was folded into three layers to have the area fitted on the circle area before measuring. Three different areas of a specimen were measured at illuminant D65 and 10° observer angle to obtain the CIE tristimulus values (Y, x, and y). The three calculated values of whiteness that resulted were averaged to obtain one reading of whiteness index. Two readings of whiteness index were taken from each specimen.

The size of each fabric piece that could be finished at one time was 11x17 in., and six such pieces were required to provide sufficient fabric for all the specimens needed to measure the full set of mechanical and durable press properties. Thus, six pieces of the cotton twill fabric needed to be finished with the same BTCA, DMDHEU, or BTCA/IA/AA combinations. There were one BTCA formulation, one DMDHEU formulation, and 32 finish formulations of BTCA/IA/AA combinations total. The six pieces of fabric were considered one sample of each finishing formulation. The measured fabric properties all

required different sizes and numbers of specimens, as described above. The cutting patterns in Figures 2 to 5 show how specimens were taken from six different pieces of fabric for measuring the fabric properties. The specimen dimensions shown in the figures are one-third the size of the actual dimensions.

Experimental Design

Good durable press finishing of cotton fabric to impart wrinkle recovery, provide a high durable press rating, and give low shrinkage and low loss of tensile strength and abrasion resistance depends, in general, on the durable press chemical crosslinking agents, the catalysts, the additives used in the impregnation bath, and the curing time and temperature. Peterson (1983) used the fractional factorial experimental design to study the relationship between the finishing conditions and the resulting fabric performance characteristics, including formaldehyde release from formaldehyde reactants. In his study, the influence factors, the finishing variables, were the concentration of the durable press chemical agent, the catalyst ratio, and the curing time. The main analysis in the present research had the purpose of determining the effects of BTCA/IA/AA combinations and several other durable press finishing variables on key properties of the 3/1 cotton twill finished fabric: the mechanical properties of tear strength and breaking strength, the durable press properties measured by durable press ratings and wrinkle recovery angle, and whiteness. These key properties were the dependent variables in the statistical analysis. In the statistical analysis, regression equations relating the fabric finish conditions to the dependent variables were estimated by the SAS computer program using multiple linear regression analysis.

The amount of potassium persulfate initiator, the drying temperature and time, and the curing temperature were fixed influence factors that were used in all the finishing of specimens with BTCA/IA/AA combinations. Based on the wrinkle recovery angle and breaking strength results of the fabric specimens finished with several candidate PCA combinations in the preliminary analysis, the amount of 1.5% o.w.m. of potassium persulfate initiator, the drying conditions of 100 °C for 10 min., and the curing temperature of 180 °C were suitable for driving the polymerization reaction of PCA combinations to occur in a short time and to impart good wrinkle recovery angle. Note that the curing temperature of 180 °C is the most widely used in research on nonformaldehyde DP finishing of cotton due to the better results on durable press properties than those obtained with other curing temperatures (Andrews, 1990; Mehra & Mehra, 1991; Reinhardt, Bhattacharyya, Sahasra-budhe & Mistry, 1994; Welch & Andrews, 1989 a&b; 1990). The concentration of BTCA, the concentration of IA, the concentration of AA, the mole ratio of monomers to the sodium hypophosphite monohydrate catalyst, and the curing time were the variable influence factors in the BTCA/IA/AA finishing, which were the independent variables in the regression analysis. Specifically, these were (a) two concentrations, 2% and 3% (o.w.b.), of BTCA, (b) two concentrations, 6.4% and 9.6% (o.w.b.), of IA, (c) two

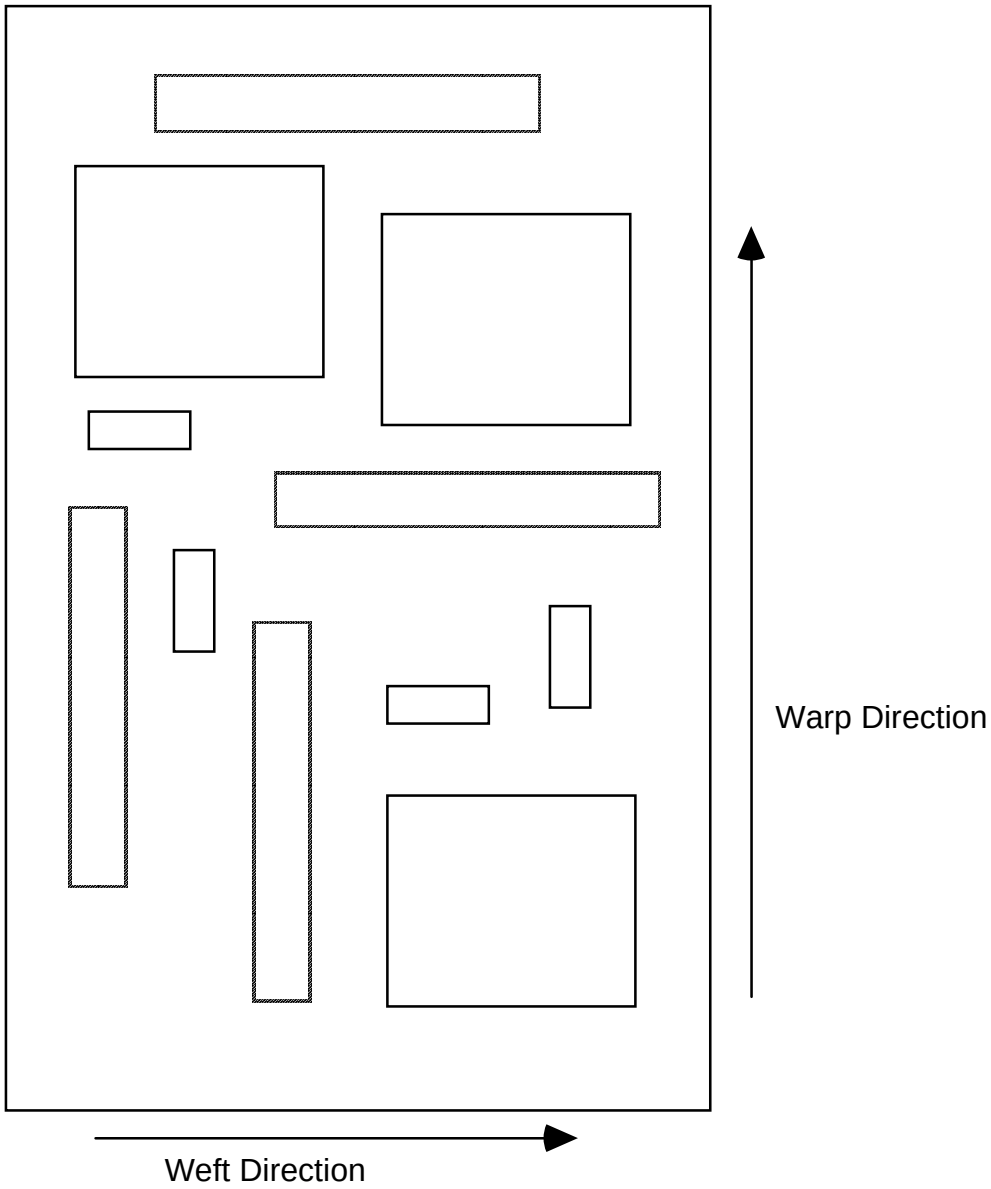
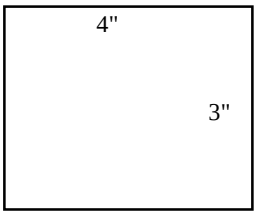


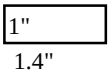
Figure 2. Cutting pattern, in the first, of the six fabric pieces finished under a common set of conditions, for specimens to measure breaking strength, tear strength, and wrinkle recovery angle.

Note.

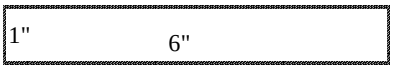
Specimen Dimensions



Tear Strength



Wrinkle Recovery Angle



Breaking Strength

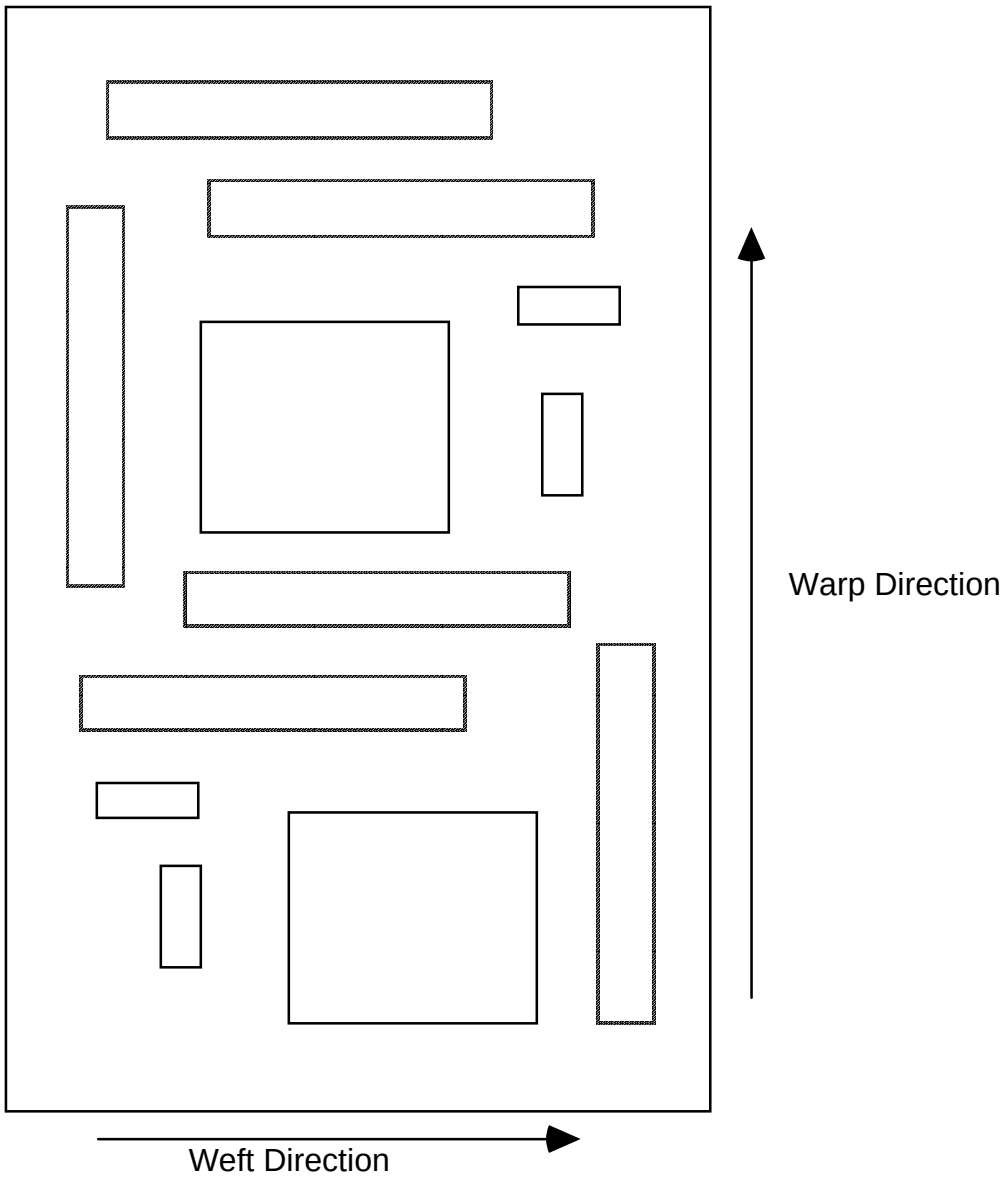
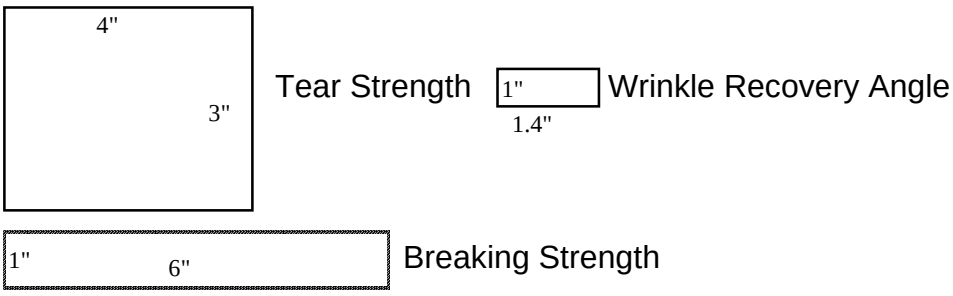


Figure 3. Cutting pattern, in the second of the six fabric pieces finished under a common set of conditions, for specimens to measure breaking strength, tear strength, and wrinkle recovery angle.

Note.

Specimen Dimensions



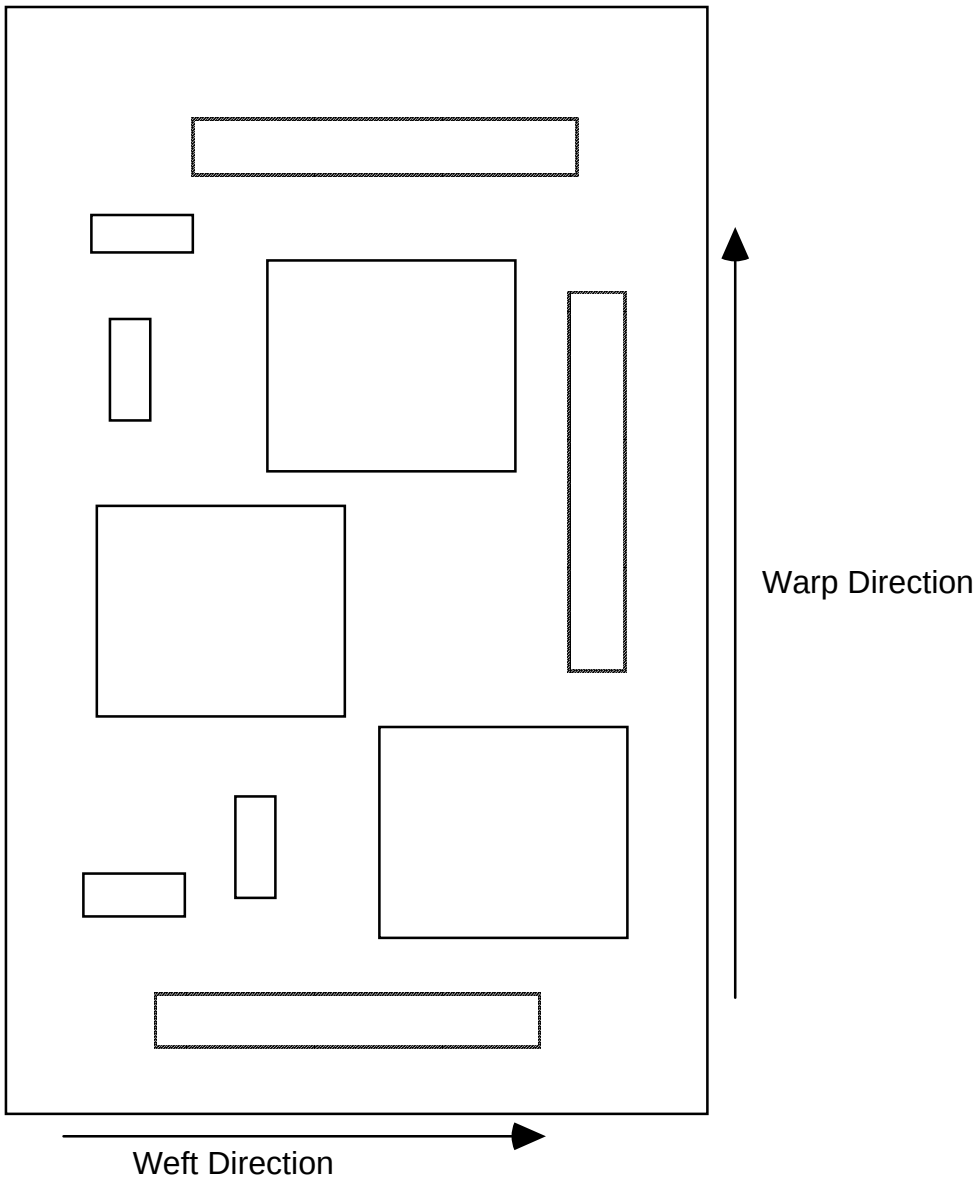
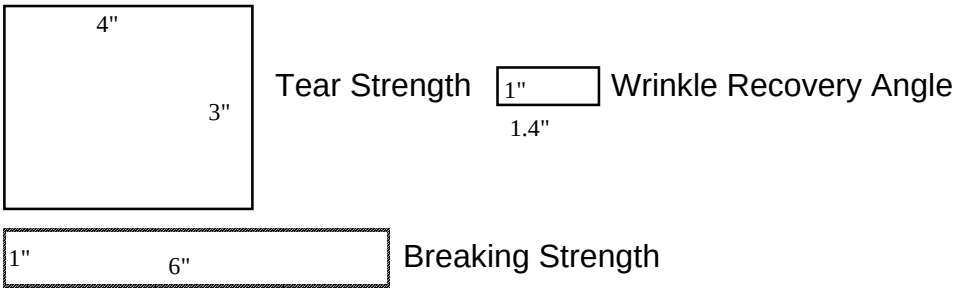


Figure 4. Cutting pattern, in the third of the six fabric pieces finished under a common set of conditions, for specimens to measure breaking strength, tear strength, and wrinkle recovery angle.

Note.

Specimen Dimensions



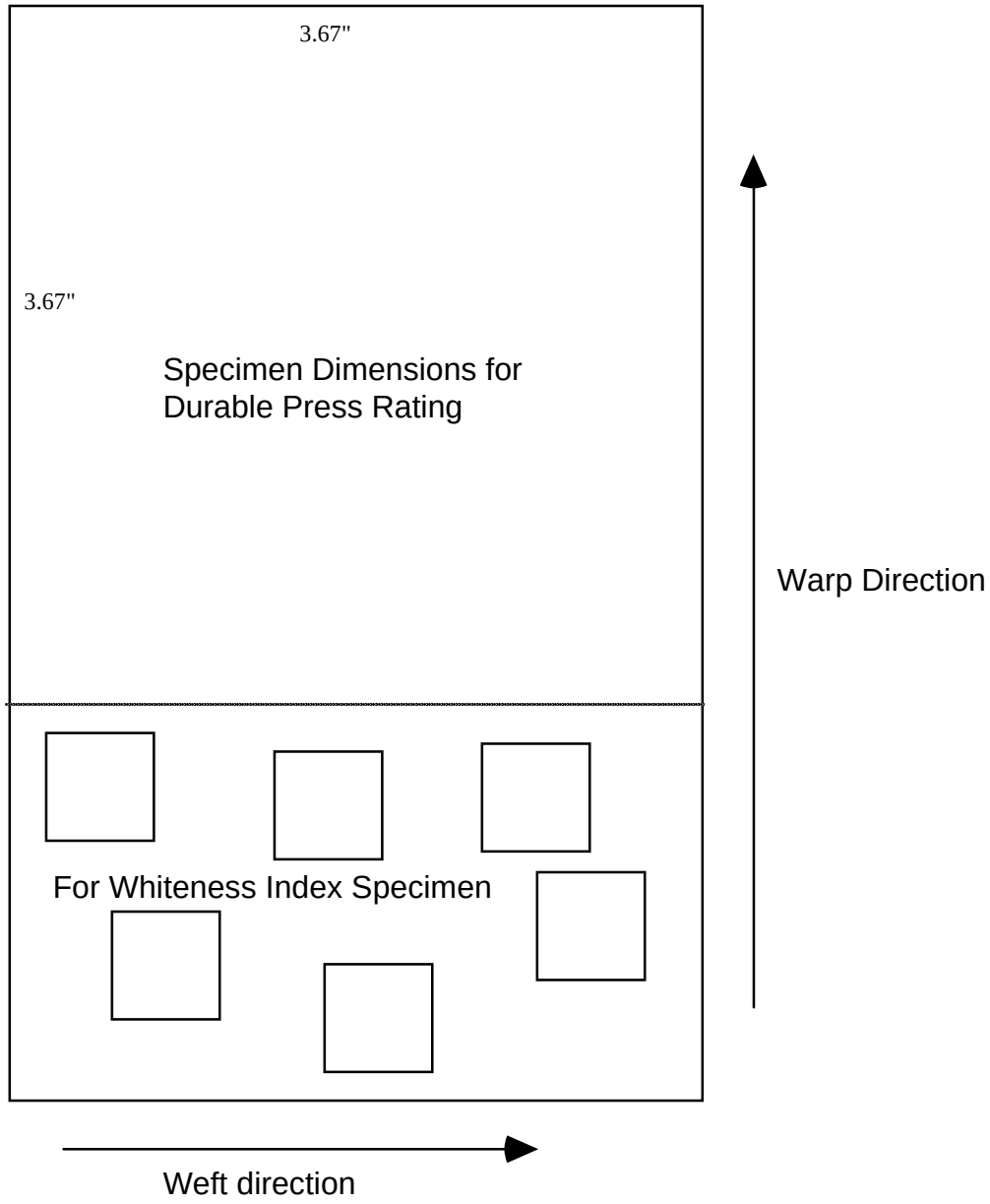


Figure 5. Cutting pattern, in the fourth, fifth, and sixth of the six fabric pieces finished under a common set of conditions, for specimens to measure durable press rating and whiteness index.

Note. Specimen Dimensions

Whiteness Index

concentrations, 1.77% and 3.54% (o.w.b.), of AA, (d) two mole ratios, 1:1 and 1:0.8, of the monomers to the sodium hypophosphite monohydrate catalyst, and (e) two curing times, 90 sec. and 3 min., at 180 °C. As seen, each influence factor had two levels. A total of 32 finishing treatments was given to fabric specimens in the BTCA/IA/AA combinations. The 32 came from 2^5, where the 2 was the level of each influence factor and the 5 was the total number of studied influence factors.

The breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press ratings after one and five launderings of finished cotton fabric specimens were the mechanical and durable press properties evaluated in this research. These five properties were measured on cotton fabrics finished with different BTCA/IA/AA combinations. The first four properties were measured on the unwashed finished fabric specimens. The durable press ratings were made on the finished fabric specimens after one and five launderings. The tested properties were as follows:

Y_1 = breaking strength

Y_2 = tear strength

Y_3 = wrinkle recovery angle

Y_4 = whiteness index

Y_5 = durable press rating

Data Analysis

The data analysis section describes three types of statistical analysis. The first concerns the means and the standard deviations of each tested property: breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press rating. The next discusses the regression analysis that involves the regression with a full model of each property and the simplification of the full model for each tested property. The last section deals with the comparison, using t-Test, of the tested properties of the fabric specimens finished with the selected BTCA/IA/AA combinations with those finished with either the BTCA or DMDHEU reactant.

Means and standard deviations. As a way to summarize and examine the data, the means and standard deviations obtained for each measured fabric property are reported in several tables in the Results and Discussion chapter. The general concept of means is the sum of the measurements divided by the total number of measurements; the way of reporting the means for wrinkle recovery angle in this study does not exactly follow this definition. Each mean for wrinkle recovery angle is the sum of the average wrinkle recovery angles in the warp and weft directions, which is the way that means for this property are normally reported in textile research publications. The means for the other properties follow the above definition of mean.

Regression analysis. The goal of regression analysis is to develop an equation that can be used to predict the dependent variable (Y) based on independent variables (X_1, X_2,..., or X_n). For this study, the regression analysis was used to predict breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press rating. The independent variables were

concentrations of BTCA, IA, and AA; mole ratios of monomers to catalyst; and curing times.

The regression analysis involved two steps. The first step was to run a full model with all the independent variables. The full model for each dependent variable is described in detail later in this section. The results of running the regression analysis of the full model provided the R^2 of the model, the coefficient estimates, and the significance level of each variable. The R^2 is the proportion of the variability in the dependent variable predicted by the independent variables. The coefficient estimates indicate the change in the dependent variable for a one-unit change in the independent variable. The significance level indicates the probability of an F-test that large by chance. The .05 level was considered a significant level this study. The next step of the regression analysis was to simplify the full model by using the backward elimination procedure and maximum R^2 procedure. The reason for conducting these two procedures was to find a potentially simpler model that could predict the dependent variable nearly as effectively as the full model did.

The backward elimination procedure eliminates the least significant variables one by one from the full model until every variable left in the model is significant at the .10 level. The last model obtained from the backward elimination procedure was run through the maximum R^2 procedure. This procedure indicated how R^2 and Mallows C_p are affected by the number of variables in the model. The Mallows C_p is defined as a prediction value that estimates the model. It attempts to estimate the total error between variance (over-fitting error) and bias (under-fitting error). A small C_p is good, but it should be close to the number of the parameters plus one. The results from the maximum R^2 procedure were used to plot a linear graph of the number of variables in the model against the R^2 and C_p values. Based on the graph obtained from the maximum R^2 procedure, a model was selected to have a high R^2 and low C_p. Predicted values from the three regression models--the full model, the high R^2 model, and the low C_p model--were compared to the actual value of the property obtained from the actual measurements on the fabric finished with BTCA/IA/AA combinations. The bar graphs of these demonstrate how well the predicted values obtained from the regression equations match the actual measurement for each property.

The multiple regression model relating one dependent variable (Y) to a set of quantitative independent variables (X_1, X_2,..., or X_n) is a direct extension of a polynomial regression model in one independent variable (Ott, 1992). The regression equation model can be a linear model, a quadratic model, etc. Since there were two levels of each independent variable in this study, the maximum power of the independent variable could only be 1. The maximum power is the number of levels minus 1. The regression model in this study could only be a linear model (one power). The category variable did not abide by this rule. The category variable is called the “dummy variable” in regression analysis. The number of dummy variables is equal to the number of categories minus 1. The

dummy variable represents a shift in the intercept in the case of no interaction, and a shift in the slope in the case of interaction.

The full model implied that all possible variables had to be included in the model. All possible variables could be the individual independent variables, the category or dummy variable, and their interaction terms. The interaction terms were added in the full model because each independent variable may enhance or deteriorate the ability of each other to act on the mechanical and durable press properties and whiteness of finished fabric specimens. Specifically, the reactants do not form effective ester linkages with cellulose hydroxyl groups without the proper catalyst and curing condition.

The full model for each tested property in this study was not the same because each tested property did not have the same number of categories; the full model could not be used identically for the five tested properties. The categories for this study were the direction of the specimen tested, the times laundered, and the different observers. The importance of the categories is exemplified by the results in the preliminary analysis. According to the measurement results on the wrinkle recovery angle and breaking strength of the finished fabric specimens in the preliminary analysis, each of those two properties differed by fabric direction in the specimens finished with the 3%BTCA/6.4%IA/1.77%AA combination. For the breaking strength, there were significant differences, $\underline{p}$=.0001, in the warp and weft directions; the breaking strength was higher in the warp direction than in the weft. For the wrinkle recovery angle, there were significant differences, $\underline{p}$=.0001, in the four fabric directions of warp face to face, warp back to back, weft face to face, and weft back to back. The wrinkle recovery angles in the directions of warp back to back and weft face to face were similar and were larger than in the other two directions. The warp face to face direction had the smallest wrinkle recovery angles. The full model for each property is described next.

The full model for the breaking strength and tear strength needed to be considered in regard to whether they were measured in the warp or weft direction of the fabric. The load distribution among the fibers and the fiber extendibility may not be the same in specimens cut from different directions. The full model used in the study for breaking strength or tear strength was as follows:

$$Y = \beta_0 + \sum_{i=1}^{6}\left(\beta_i X_i + \sum_{j=1}^{6}\beta_{ij} X_i X_j\right) + E \qquad (1)$$

where Y = a dependent variable; breaking or tear strength

X's = the independent variables, called regressors, are as follow

X_1 = 2% or 3% o.w.b. concentration of BTCA

X_2 = 6.4% or 9.6% o.w.b. concentration of IA

X_3 = 1.77% or 3.54% o.w.b. concentration of AA

X_4 =1, if 1:1 mole ratio of monomers to catalyst;
0.8, if 1:0.8 mole ratio of monomers to catalyst

X_5 = 1.5, if curing time was 90 sec.; 3, if curing time was 3 min.

X_6 = 1 for warp direction, indicating the regression equation was for the breaking strength or the tear strength in the warp direction;

0 for weft direction, indicating the regression equation was for the breaking strength or the tear strength in the weft direction

ß's = regression coefficients to be estimated

E = error term in which all deviations were compiled that could not be described by X_1 to X_6

The full model for wrinkle recovery angle needed to be considered in regard to whether the angle was measured in the warp or weft direction and whether the fabric was folded face to face or back to back for each direction. The wrinkle recovery angle of each specimen was taken from four different directions, warp back to back, warp face to face, weft back to back, and weft face to face. The dummy variables were 3 because the number of directions was 4 minus 1. The dummy variables X_6, X_7, and X_8 were added in the equation for the wrinkle recovery angle. The full model for the wrinkle recovery angle property used in the study follows:

$$Y = \beta_0 + \sum_{i=1}^{8}\left(\beta_i X_i + \sum_{j=1}^{8}\beta_{ij} X_i X_j\right) + E \qquad (2)$$

where Y = a wrinkle recovery angle for one of each of four directions depending on whether the three dummy variables were replaced with 0 or 1 in the combination of three dummy variables

X's = the independent variables, called regressors, are as follow

X_1 = 2% or 3% o.w.b. concentration of BTCA

X_2 = 6.4% or 9.6% o.w.b. concentration of IA

X_3 = 1.77% or 3.54% o.w.b. concentration of AA

X_4 = 1, if 1:1 mole ratio of monomers to catalyst;
0.8, if 1:0.8 mole ratio of monomers to catalyst

X_5 = 1.5, if curing time was 90 sec.;3, if curing time was 3 min.

X_6, X_7, and X_8 = 0 or 1 as shown in the combinations below:

	The Dummy Variables		
	X_6	X_7	X_8
Warp back to back	0	0	0
Warp face to face	1	0	0
Weft back to back	0	1	0
Weft face to face	0	0	1

ß's = regression coefficients to be estimated

E = error term in which all deviations were compiled that could not be described by X_1 to X_8

The full model for the whiteness index did not need consideration of the direction effect. The whiteness was measured only on the face of the specimens of the 3/1 cotton twill fabric; therefore, no dummy variable was added in the full model. The full model for whiteness index used in the study follows:

$$Y = \beta_0 + \sum_{i=1}^{5}\left(\beta_i X_i + \sum_{j=1}^{5}\beta_{ij} X_i X_j\right) + E \qquad (3)$$

where Y = whiteness index

X's = the independent variables, called regressors, are as follow

X_1 = 2% or 3% o.w.b. concentration of BTCA
X_2 = 6.4% or 9.6% o.w.b. concentration of IA
X_3 = 1.77% or 3.54% o.w.b. concentration of AA
X_4 = 1, if 1:1 mole ratio of monomers to catalyst;
0.8, if 1:0.8 mole ratio of monomers to catalyst
X_5 = 1.5, if curing time was 90 sec.; 3, if curing time was 3 min.
ß's = regression coefficients to be estimated
E = error term in which all deviations were compiled that could not be described by X_1 to X_5

The full model for the durable press rating needed consideration in regard to who performed the rating and whether the rating was after one or five launderings. The standard test method for durable press rating requires three observers to rate each specimen; two dummy variables were used in the regression model to account for the observer effect. In addition, the durable press rating of each specimen was made twice: the first rating after a specimen was washed one time and the second rating after that same specimen was rewashed four times. A dummy variable was used in the regression model to account for the one and five launderings. The full model for the durable press ratings in the study follows:

$$Y = \beta_0 + \sum_{i=1}^{8}\left(\beta_i X_i + \sum_{j=1}^{8}\beta_{ij} X_i X_j\right) + E \qquad (4)$$

where Y = the durable press rating (either one or five launderings)
X's = independent variables, called regressors, are as follow
X_1 = 2% or 3% o.w.b. concentration of BTCA
X_2 = 6.4% or 9.6% o.w.b. concentration of IA
X_3 = 1.77% or 3.54% o.w.b. concentration of AA
X_4 = 1, if 1:1 mole ratio of monomers to catalyst;
0.8, if 1:0.8 mole ratio of monomers to catalyst
X_5 = 1.5, if curing time was 90 sec.; 3, if curing time was 3 min.
X_6 = 0 for first laundering, 1 for fifth launderings
X_7, and X_8 = 0 or 1 as shown in the combinations below:

	The Dummy Variables	
	X_7	X_8
First observer	0	0
Second observer	1	0
Third observer	0	1

ß's = regression coefficients to be estimated
E = error term in which all deviations were compiled that could not be described by X_1 to X_8

Test trials were conducted after regression analysis had been done. The test trials were to determine how well the three regression equation models of each of five properties--the full model, the high R^2 model, and the low C_p model--predicted the results obtained by measuring the properties in four different test trial finish formulations. In both Trials 1 and 2, each variable had a value in the

range between the high and low levels of each variable that was used to build the regression equations. In Trial 3, the concentration of AA was equal to 5.31% o.w.b. which was far beyond the studied range. Trial 4 had the concentration of IA (12.8% o.w.b.) and AA (5.31% o.w.b.) outside the range, while the values of the other variables in Trials 3 and 4 were used in the studied range. The finish formulations of the four test trials are as follows.

Test trial #1. All variables used in the finish formulation were in the original range, for generating the data for estimating the equations. The finish formulation was 2.5% BTCA, 8% IA, 2.95%AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1 mole ratio of monomers to the catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate. The finish application was followed by predrying at 100 °C for 10 min. and curing at 180° C for 2 min.

Test trial #2. All variables were in the original range. The finish formulation was 3% BTCA, 9.6% IA, 2.65%AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:0.9 mole ratio of monomers to the catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate. The finish application was followed by predrying at 100 °C for 10 min. and curing at 180 ° C for 2 min.

Test trial #3. The AA variable was outside the original range. The finish formulation was 3% BTCA, 9.6% IA, 5.31%AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:1 mole ratio of monomers to the catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate. The finish application was followed by predrying at 100 °C for 10 min. and curing at 180 ° C for 2 min.

Test trial #4. IA and AA variables were out of the studied range. The finish formulation was 2.5% BTCA, 12.8% IA, 5.31%AA, 1% polyolefin emulsion, 0.2% Triton X-100, 1:0.8 mole ratio of monomers to catalyst sodium hypophosphite monohydrate, and 1.5% o.w.m. initiator potassium persulfate. The finish application was followed by predrying at 100 °C for 10 min. and curing at 180 °C for 3 min.

For testing research hypotheses 1 to 3, the regression equation with the high R^2 was used. Most researchers normally use high R^2; rather than low C_p, to judge model fit. The smaller model such as the regression with high R^2 is used more easily for interpreting the results than is the full model because it contains fewer variables and most variables in the model are significant. Research hypotheses 1 to 3 address the condition that the ability of each independent variable, BTCA, IA, and AA concentrations, mole ratio of monomers to catalyst, and curing time, will be dependent on each other to affect the fabric properties finished with BTCA/IA/AA formulations. If the regression with the high R^2 contains any interaction terms of the main independent variables, those hypotheses can be accepted.

Comparison of measured properties of the fabric finished with the selected BTCA/IA/AA combinations with those of the fabric finished with BTCA only or with DMDHEU. The same set of five properties was measured on fabric finished with either BTCA or DMDHEU and on fabric finished with BTCA/IA/AA

combinations. The results of the five tested properties on the fabric specimens finished with BTCA or with DMDHEU are the standard that this study attempts to achieve from fabric finishing with BTCA combinations. After obtaining fabric-property results from the 32 finish formulations of BTCA/IA/AA combinations, a selected BTCA/IA/AA combination that provided properties comparable to those of fabric specimens finished with either BTCA or DMDHEU reactant was used for conducted a pair-comparison by t-Test. The selected BTCA/IA/AA formulation that was used for comparison with either BTCA or DMDHEU could be or could not be used as the same BTCA/IA/AA formulation. The t-Test is a statistical method that allows comparison between the mean value of each of five tested properties (breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press rating) of fabric specimens finished with a selected BTCA/IA/AA formulation and the mean value of the same property obtained with the BTCA or the DMDHEU finish. According to the result of t-Test, the null hypothesis to be tested that the mean value of a fabric property obtained from the BTCA/IA/AA combination treatment is equal to the mean value of the same property obtained from either the BTCA or DMDHEU treatment. If two means are significantly different (p-value smaller than .05), the null hypothesis is rejected. The overall results from the t-Test procedure indicate whether the measured properties of fabric specimens finished with the selected BTCA/IA/AA formulation are or are not significantly different from those of fabric specimens finished with either BTCA or DMDHEU reactants by looking at the p-value for answering the research hypotheses 4 and 5 that were stated in Chapter III.

CHAPTER V
Results and Discussion

This chapter contains results and discussion of the descriptive statistics involving means and standard deviations and of the data analysis by regression for the five measured fabric properties: breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press rating. This chapter also includes the comparison of the actual measurements of the fabric properties of specimens finished with BTCA/IA/AA combinations with the values of those properties as predicted by the estimated regression equations, and the comparison, using t-Test, of the fabric properties of specimens finished with the BTCA/IA/AA combinations with the fabric properties of specimens finished with the BTCA or with the DMDHEU reactant.

Means and Standard Deviations of Fabric Properties

Breaking-Strength Fabric Property

Breaking strength values were not converted to the breaking strength retentions of the fabric specimens for calculating the means and standard deviations. The breaking strength property is reported as the force in kilograms to break the specimens either in the warp or the weft direction, and is rounded up to the nearest hundredth. The means and standard deviations of this property are for five specimens each, in the warp direction, that had been finished with 32 BTCA/IA/AA formulations. Table 9 summarizes the means and standard deviations of the breaking strength in the warp direction. According to the mean values of the breaking strength of fabric specimens in the warp direction shown in Table 9, it can be concluded that when the other variables were kept constant, increasing curing time, increasing BTCA concentration, or increasing IA concentration affected the warp-direction breaking strength in a trend of decreasing this property. Increasing the BTCA concentration from 2% to 3% o.w.b. affected the breaking strength in the range of -0.71 to +0.48. Increasing the IA concentration from 6.4% to 9.6% o.w.b. affected this property in the range of -0.82 to +0.51. Increasing curing time from 90 sec. to 3 min. affected this property in the wider range of -1.09 to +0.40. On the other hand, when those other finishing variables remained constant, increasing the AA concentration from 1.77% to 3.54% o.w.b. or the mole ratio of monomers to catalyst from 1:0.8 to 1:1 affected the breaking strength in the warp direction of the finished fabric specimens in a trend of improvement. Increasing the AA concentration affected the property in the range of -0.19 to +0.93. Increasing the mole ratio of monomers to catalyst improved the breaking strength in the range of +0.40 to +1.11. By comparing the dispersion of the ranges obtained with each finishing variable, it can be concluded that the order of independent variables from greatest to least improvement in the breaking strength of the finished fabric specimens for the warp direction appears to be: mole ratio of monomers to catalyst > AA concentration > BTCA concentration > IA concentration > curing time.

Table 9 Means and standard deviations (in parentheses) of the breaking strength (kg) in the warp direction of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	6.23 (0.80)	5.41 (0.71)	6.30 (0.64)	5.86 (0.46)
3.54%AA	6.30 (0.42)	6.34 (0.76)	6.35 (0.59)	5.92 (0.38)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	6.88 (0.33)	6.38 (0.38)	6.55 (0.27)	6.27 (0.45)
3.54%AA	6.72 (0.41)	6.79 (0.29)	6.39 (0.36)	6.08 (0.70)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	5.14 (0.63)	5.50 (0.69)	5.62 (0.11)	5.49 (0.43)
3.54%AA	5.64 (0.35)	5.61 (0.15)	5.64 (0.31)	5.78 (0.64)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	6.12 (0.41)	6.26 (0.36)	6.09 (0.31)	6.60 (0.29)
3.54%AA	6.57(0.16)	6.24 (0.60)	6.26 (0.61)	6.47 (0.33)

Table 10 summarizes the mean and standard deviations of the breaking strength in the weft direction. The means and standard deviations of this property are for eight specimens each, in the weft direction, that had been finished with 32 BTCA/IA/AA formulations. According to the mean values of the breaking strength of the fabric specimens in the weft direction, increasing the BTCA concentration from 2% to 3% o.w.b., when the other finishing variables remained constant, affected the breaking strength property in the range of -0.48 to -0.01. When the BTCA concentration increased, only 3%BTCA/9.6%IA/3.54%AA with 1:0.8 mole ratio of monomers to catalyst and 3-min. curing provided better breaking strength in the weft direction, by +0.1 unit, than did 2%BTCA/9.6%IA/3.54%AA with the same treatment condition. Increasing the IA concentration from 6.4% to 9.6% o.w.b. affected the breaking strength in the range of -0.45 to +0.14. Increasing the curing time from 90 sec. to 3 min. decreased the breaking strength of the finished fabric in the range of -0.54 to -0.01. By comparing the dispersion of the ranges obtained with the above three finishing variables, it was concluded that increasing BTCA concentration, increasing IA concentration, or increasing curing time affected the breaking strength in the weft direction in a trend of decreasing this property. On the other hand, increasing the AA concentration from 1.77% to 3.54% o.w.b. or the mole ratio of monomers to catalyst from 1:0.8 to 1:1 affected this property in a trend of improvement in the ranges of -0.24 to +0.47, and -0.29 to +0.36, respectively.

Based on the observed ranges of the breaking strength in the weft direction with respect to finishing variables as just described, the order of the independent variables from greatest to least improvement the breaking strength appears to be: AA concentration > mole ratio of monomers to catalyst > IA concentration > BTCA concentration > curing time. The order of the independent variables from the most to the least improvement in the breaking strength of the finished fabric specimens was not the same order for both the warp and weft directions; however, it can be concluded that increased mole ratio of monomers to catalyst or increased AA concentration tended to improve the breaking strength property, but increased BTCA concentration, IA concentration, or curing time tended to decrease the breaking strength.

Tear-Strength Fabric Property

The fabric tear strength was measured as the percentage of the total force of 3200 g., which was used for testing. To obtain the tear resistance (i.e., tear strength) of a specimen, the percentage was converted to the gram-force by multiplying the percentage by 32 to get the total force in grams. The tear strength was not converted to the tear strength retention for calculating the means and standard deviations of tear strength fabric property. The mean values of tear strength of fabric specimens finished with 32 BTCA/IA/AA formulations are reported as the gram-force and rounded up to whole numbers. The standard deviations of the tear strength in the warp direction were the variation over four fabric specimens finished with each BTCA/IA/AA formulation. Table 11 summarizes the means and standard deviations of the tear strength in the warp direction of the fabric specimens finished with 32 formulations.

Table 10 Means and standard deviations (in parentheses) of the breaking strength (kg) in the weft direction of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	4.68 (0.14)	4.60 (0.18)	4.41 (0.13)	4.26 (0.18)
3.54%AA	4.61 (0.12)	4.60 (0.34)	4.17 (0.24)	4.23 (0.23)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	4.76 (0.21)	4.31 (0.20)	4.50 (0.27)	4.30 (0.15)
3.54%AA	4.84 (0.19)	4.78 (0.41)	4.41 (0.37)	4.37 (0.26)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	4.22 (0.31)	4.11 (0.13)	4.04 (0.20)	3.99 (0.15)
3.54%AA	4.25 (0.26)	4.09 (0.22)	4.06 (0.17)	4.19 (0.21)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	4.44 (0.26)	4.30 (0.22)	3.96 (0.21)	4.10 (0.12)
3.54%AA	4.61(0.27)	4.33 (0.21)	4.22 (0.29)	4.10 (0.28)

Table 11 Means and standard deviations (in parentheses) of the tear strength (g) in the warp direction of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1268 (54.45)	1204 (47.77)	1164 (35.48)	1068 (15.32)
3.54%AA	1344 (67.88)	1260(54.45)	1188 (27.32)	1184 (55.42)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1312 (135.76)	1152 (45.25)	1192 (84.16)	1152 (45.25)
3.54%AA	1296 (109.30)	1368 (65.97)	1312 (64.00)	1368 (53.06)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1100 (54.45)	1012 (44.06)	980 (15.31)	928 (39.19)
3.54%AA	1344 (67.88)	1064 (30.64)	1032 (61.96)	988 (32.98)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1040 (66.61)	1088 (36.95)	968 (30.64)	944 (55.42)
3.54%AA	1176 (40.26)	1080 (30.67)	1056 (26.13)	976 (41.31)

According to the results shown in Table 11 for the average of the tear strength in the warp direction of the fabric specimens finished with each of 32 BTCA/IA/AA formulations, increasing the BTCA concentration affected the tear strength in the range of -312 to +16. Increasing the IA concentration affected the property in the range of -280 to +72. Increasing the curing time decreased the tear strength in the range of -288 to 0. The above three variables were the main contributors to a decrease in tear strength, whereas the AA concentration and the mole ratio of monomers to catalyst were the main contributors to an improvement of the tear strength. Increasing the AA concentration tended to improve tear strength in the range of -8 to +244. Increasing the mole ratio of monomers to catalyst also tended to improve the property in the range of -168 to +124. According to the tear strength dispersion of the ranges of each finishing variable, the order of the independent variables from the most to the least improvement in the tear strength in the warp direction appears to be: AA concentration > mole ratio of monomers to catalyst > IA concentration > BTCA concentration > curing time.

Table 12 summarizes the means and standard deviations of the tear strength in the weft direction of fabric specimens finished with BTCA/IA/AA combinations. The means and standard deviations of the tear strength in the weft direction reported in Table 12 were the variation over four fabric specimens finished with each BTCA/IA/AA formulation. According to the results of the average tear strength (in weft direction) of fabric specimens finished with each of the BTCA/IA/AA formulations in Table 12, increasing the BTCA concentration affected the tear strength in the range of -248 to +12. Increasing the IA concentration affected the property in the range of -200 to +32. Increasing the curing time decreased the tear strength in the range of -188 to 0, but increasing the AA concentration tended to improve the property in the range of -24 to +288. Increasing the mole ratio of monomers to catalyst also tended to improve the tear strength in the range of -160 to +88. According to the tear strength dispersion of the ranges of each finishing variable, the order of the independent variables from the most to the least improvement in the tear strength in the weft direction appears to be: AA concentration > mole ratio of monomers to catalyst > IA concentration > BTCA concentration > curing time. The order of the finishing variables from the most to the least improvement in the tear strength was the same in both the warp and the weft directions.

Wrinkle-Recovery-Angle Fabric Property

Table 13 summarizes the summed means of the wrinkle recovery angles (in degrees) for the warp and weft directions of the specimens finished with the 32 formulations. The means of the summed average wrinkle recovery angles in both directions are reported and rounded up to whole numbers. The standard deviation values were calculated from the variation over twelve measurements on the fabric specimens finished with each of the 32 BTCA/IA/AA formulations. According to the results shown in Table 13 for the 32 finish formulations, increasing the concentration of BTCA from 2% to 3%, while the other finishing variables remained constant, increased the wrinkle recovery angles of the finished fabrics in the range of +4° to +28°. Increasing the concentration of IA

Table 12 Means and standard deviations (in parentheses) of the tear strength (g) in the weft direction of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1272 (30.64)	1176 (27.71)	1192 (27.71)	1112 (16.00)
3.54%AA	1312 (26.13)	1212 (20.13)	1200 (34.56)	1184 (22.63)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1232 (76.18)	1264 (18.48)	1212 (56.00)	1156 (27.32)
3.54%AA	1280 (45.25)	1240 (48.00)	1224 (70.96)	1200 (22.63)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1084 (37.81)	1064 (27.71)	1016 (48.00)	1020 (27.32)
3.54%AA	1312 (26.13)	1112 (9.24)	1064 (46.19)	1060 (20.13)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	1088 (52.26)	1112 (30.64)	1088 (45.25)	1056 (0.00)
3.54%AA	1152 (73.90)	1096 (30.64)	1100 (51.22)	1108 (35.48)

Table 13 Means and standard deviations of the wrinkle recovery angles (°) in the warp and weft directions of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	259° (11.24)	267° (10.47)	266° (10.80)	278° (8.17)
3.54%AA	256° (11.66)	265° (10.46)	263° (10.05)	272° (9.60)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	255° (8.87)	269° (9.48)	271° (8.79)	276° (8.07)
3.54%AA	257° (8.20)	267° (8.96)	264° (9.95)	272° (11.26)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	269° (6.96)	273° (8.87)	273° (7.82)	280° (7.98)
3.54%AA	267° (10.76)	278° (7.64)	275° (9.57)	282° (9.27)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	267° (8.27)	276° (8.07)	273° (9.31)	285° (8.38)
3.54%AA	252° (9.68)	276° (9.32)	280° (7.77)	284° (10.13)

from 6.4% to 9.6%, while the other finishing variables were kept constantly, also increased the wrinkle recovery angles in the range of +4° to +24°. Increasing the concentration of AA from 1.77% to 3.54% either increased or decreased the wrinkle recovery angles of the finished fabric specimens, depending on the finish formulations. Increasing the concentration of AA in the finish combination of 2%BTCA/6.4%IA/AA with 1:1 mole ratio of monomers to catalyst cured for 3 min. caused the greatest decrease in the average wrinkle recovery angle (-15°), whereas increasing the concentration of AA in the other finish combinations affected the wrinkle recovery angles in the range of -3° to +7°. Increasing the mole ratio of monomers to catalyst in the finish formulation of 2%BTCA/6.4%IA/3.54%AA combination cured for 3 min. decreased the wrinkle recovery angle, by -15°, of the finished fabric specimens, which was the greatest change with any of the treatment combinations; increasing the mole ratio of monomers to catalyst in the other finish formulations affected the wrinkle recovery angles in the range of -4° to +2°. Increasing the curing time from 90 sec. to 3 min. contributed to an increase in the wrinkle recovery angle in the range of +2° to +16°. One finish combination, that of 2%BTCA/6.4%IA/3.54%AA with 1:1 mole ratio of monomers to catalyst cured for 3 min., provided 5° fewer degrees than that of the same treatment cured for 90 sec.

Based on the observed ranges of the wrinkle recovery angles with respect to each finishing variable, as just described, the order of the independent variables from the greatest to least effect on the wrinkle recovery angles appears to be: BTCA concentration > IA concentration > curing time > AA concentration > mole ratio of monomers to catalyst.

Whiteness-Index Fabric Property

A summary of the means and standard deviations of the whiteness index of the fabric specimens finished with 32 BTCA/IA/AA formulations is reported in Table 14. The mean values of the whiteness index were rounded up to whole numbers. The means and standard deviations of this property were the variation over six readings of the whiteness index of fabric specimens finished with each of 32 BTCA/IA/AA combinations.

According to the means of the whiteness index in Table 14, increasing the BTCA concentration reduced the whiteness index in the range of -6 to 0. Increasing the IA concentration decreased the whiteness index in the range of -6 to 0. Only one finish formulation, that of 3%BTCA/9.6%IA/1.77%AA with 1:0.8 mole ratio of monomers to catalyst and curing for 90 sec. increased the whiteness index by 1 unit when the IA concentration was increased from 6.4% to 9.6% o.w.b. Increasing the curing time with the finish formulation of 3%BTCA/6.4%IA/1.77%AA with 1:0.8 mole ratio of monomers to catalyst only increased the whiteness index by 2 units, whereas increasing the curing time with the other finish formulations decreased this property in the range of -6 to -1. The above three finishing variables reduced the whiteness index of the fabric specimens finished with BTCA/IA/AA formulations when those variables increased from the low level to the high level. Increasing the mole ratio of monomers to catalyst or the AA concentration improved the whiteness index of the finished fabric specimens in the range of +1 to +8 in both variables.

Table 14 Means and standard deviations (in parentheses) of the whiteness index of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	61 (2.75)	60 (1.32)	55 (4.83)	56 (1.07)
3.54%AA	65 (0.92)	63 (1.29)	63 (0.89)	62 (1.97)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	65 (0.78)	63 (0.84)	63 (1.78)	61 (1.44)
3.54%AA	69 (0.43)	66 (0.70)	69 (0.75)	67 (0.51)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	59 (1.55)	55 (1.70)	57 (0.72)	55 (0.78)
3.54%AA	62 (1.02)	62 (1.21)	62 (0.79)	56 (1.04)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	62 (2.23)	60 (1.48)	60 (1.14)	59 (1.41)
3.54%AA	65 (1.02)	63 (1.45)	63 (0.84)	63 (1.38)

Comparing the dispersion of the ranges of each finishing variable, the order of the independent variables from the most increase to the least in the whiteness index of the finished fabric specimens appears to be: AA concentration = mole ratio of monomers to catalyst > IA concentration > BTCA concentration > curing time.

Durable-Press-Rating Fabric Property

The mean value of this property is the average of nine durable press ratings after either the first or fifth launderings of fabric specimens finished with each of the 32 BTCA/IA/AA combinations. The means of the durable press rating were rounded up to the nearest tenth. The means and standard deviations were the variation of those nine ratings of specimens. Table 15 summarizes the means and standard deviations of this property of fabric specimens for one laundering. Table 16 summarizes the means and standard deviations of this property of fabric specimens for five launderings.

According to the results of the durable press rating for one laundering in Table 15, increasing the BTCA concentration improved the durable press rating in the range of 0 to +0.5. Increasing the curing time tended to improve the property in the range of 0 to +0.3. Only one finishing formulation, that of 3%BTCA/9.6%IA/3.54%AA with 1:1 mole ratio of monomers to catalyst, decreased the durable press rating of the specimens, by -0.3 units, when the curing time increased. Increasing IA concentration, AA concentration, or the mole ratio of monomers to catalyst affected the durable press rating in the ranges of -0.1 to +0.4, -0.4 to +0.2, and -0.5 to +0.3, respectively.

Comparing the dispersion of the ranges of each finishing variable, the order of the independent variables from most to least increase in the durable press rating of the finished fabric specimens appears to be: BTCA concentration > curing time > IA concentration > AA concentration > mole ratio of monomers to catalyst. From the results of the durable press rating for five launderings in Table 16, it can be concluded that the ranges of each finishing variable affecting the property when each variable increased from the low to high level were -0.3 to +0.3 for the BTCA concentration, -0.5 to +0.2 for the curing time, -0.5 to +0.1 for the AA concentration, -0.7 to +0.1 for the IA concentration, and -0.9 to +0.2 for the mole ratio of monomers to catalyst, respectively. Comparing the dispersion of the ranges for each finishing variable of the durable press rating after five launderings, the order of the independent variables from most to least increase in this property of the finished fabric specimens appears to be: BTCA concentration > curing time > AA concentration > IA concentration > mole ratio of monomers to catalyst. The order of the AA concentration and the IA concentration after the five launderings was switched for the durable press rating from that after one laundering, while others had the same in the order for both one and five launderings.

Summary of the Means and Standard Deviations of Five Fabric Properties

By comparing the dispersion of ranges of each finishing variable that also constitutes an independent variable in the statistical analysis affecting each of the five properties, the order of the independent variables from most to least in

Table 15 Means and standard deviations (in parentheses) of the first laundering of the durable press ratings of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	3.3 (0.25)	3.4 (0.17)	3.4 (0.22)	3.4 (0.17)
3.54%AA	3.2 (0.25)	3.4 (0.17)	3.5 (0.25)	3.6 (0.22)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	3.2 (0.26)	3.1 (0.22)	3.7 (0.25)	3.6 (0.17)
3.54%AA	3.1 (0.22)	3.2 (0.26)	3.5 (0.00)	3.7 (0.25)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	3.6 (0.17)	3.5 (0.00)	3.6 (0.17)	3.7 (0.26)
3.54%AA	3.5 (0.00)	3.6 (0.17)	3.5 (0.00)	3.9 (0.22)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	3.4 (0.33)	3.3 (0.25)	3.7 (0.25)	3.8 (0.26)
3.54%AA	3.4 (0.22)	3.3 (0.25)	3.5 (0.00)	3.4 (0.22)

Table 16 Means and standard deviations (in parentheses) the fifth launderings of the durable press ratings of the specimens finished with BTCA/IA/AA at different mole ratios and curing times

1:0.8 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	2.8 (0.25)	2.9 (0.17)	3.1 (0.17)	3.0 (0.00)
3.54%AA	2.8 (0.25)	2.9 (0.22)	3.0 (0.25)	2.7 (0.36)

1:1 mole ratio of monomers to catalyst; 90 sec. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	3.0 (0.00)	2.8 (0.25)	3.1 (0.17)	2.9 (0.22)
3.54%AA	3.0 (0.00)	2.3 (0.25)	3.1 (0.17)	2.5 (0.25)

1:0.8 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	3.2 (0.26)	3.1 (0.22)	3.3 (0.25)	3.3 (0.25)
3.54%AA	3.2 (0.25)	3.1 (0.42)	3.3 (0.26)	3.2 (0.35)

1:1 mole ratio of monomers to catalyst; 3 min. curing time

	2%BTCA		3%BTCA	
	6.4%IA	9.6%IA	6.4%IA	9.6%IA
1.77%AA	2.9 (0.17)	2.8 (0.25)	3.0 (0.00)	2.8 (0.35)
3.54%AA	3.0 (0.00)	2.6 (0.39)	2.9 (0.17)	2.3 (0.36)

increasing each property of the finished fabric specimens is summarized as follows.

The order of the variables from most to least in increasing the breaking strength in the warp direction of the finished fabric specimens was: mole ratio of monomers to catalyst > AA concentration > BTCA concentration > IA concentration > curing time.

The order of the variables from most to least in increasing the breaking strength in the weft direction of the finished fabric specimens was:
AA concentration > mole ratio of monomers to catalyst > IA concentration > BTCA concentration > curing time.

The order of the variables from most to least in increasing the tear strength both in the warp and in the weft direction of the finished fabric specimens was: AA concentration > mole ratio of monomers to catalyst > IA concentration > BTCA concentration > curing time.

The order of the variables from most to least in increasing the wrinkle recovery angles of the finished fabric specimens was: BTCA concentration > IA concentration > curing time >AA concentration > mole ratio of monomers to catalyst.

The order of the variables from most to least in increasing the whiteness index of the finished fabric specimens was: AA concentration = mole ratio of monomers to catalyst > IA concentration > BTCA concentration > curing time.

The order of the variables from most to least in increasing the durable press rating after one laundering of the finished fabric specimens was: BTCA concentration > curing time > IA concentration >AA concentration > mole ratio of monomers to catalyst.

The order of the variables from most to least in increasing the durable press rating after five launderings of the finished fabric specimens was: BTCA concentration > curing time > AA concentration >IA concentration > mole ratio of monomers to catalyst.

The summary of the orders of the independent variables from most to least in improving each property, as mentioned above, indicate that those five variables can be divided into two groups. The first group contains three variables: BTCA concentration, IA concentration, and the curing time. The second group contains AA concentration and mole ratio of monomers to catalyst. The variables in the first group improved the wrinkle recovery angle and the durable press rating properties, but decreased the breaking strength, tear strength, and whiteness index of the finished fabric specimens when the levels of those independent variables increased. The variables in the second group improved the breaking strength, tear strength, and whiteness index, but decreased the properties of the wrinkle recovery angle and the durable press rating of the finished fabric specimens when the levels of those independent variables increased.

Results of the Regression Analysis

Regression Analysis of the Breaking Strength

The breaking strength data used in the regression analysis were expressed in kilograms; the breaking strength retention in percentage was not used. Breaking strength and breaking strength retention would have the same statistical result in the terms of F-value and p-value, but their parameter estimates would not be the same due to the different units.

The full model with the breaking strength as the dependent variable was run through the regression procedure. The results for the root mean square error and R^2 of the full model and the significance levels of all variables are reported in Table 17. Referring to the results of p-values in Table 17, it can be concluded that the two directions of the specimens, taken from warp or weft, were significantly different in their effect on the breaking strength property of the finished fabric specimens. Based on the significance and the sign and magnitude of the coefficient on curing time, this is the main independent variable that affected the fabric breaking strength, and its effect was to a decrease this property. The other non-interacted independent variables did not affect this property significantly, but some of their interactions did so.

The interaction terms of BTCA concentration with AA concentration, with mole ratio of monomers to catalyst, and with curing time significantly affected the breaking strength. The IA concentration and the mole ratio of monomers to catalyst decreased the ability of the curing time to deteriorate the breaking strength of the finished fabric specimens. The BTCA concentration and the mole ratio of monomers to catalyst affected this property in a trend of improving it significantly in the warp direction. The full model was difficult to use for interpreting clearly what variables or their interaction terms affected this property in a trend of increasing or decreasing because it contained many significant variables and nonsignificant variables that were not necessary in the model. Therefore, the full model was simplified by running it through the backward elimination procedure.

The model obtained from the backward elimination procedure, with the goal of maximizing the R^2, for this property is reported in Appendix M. Figure 6 shows a graph of the R^2 and Mallows C_p against the number of variables in the stepwise models. This figure shows that a regression model containing just three independent variables has a good high R^2 comparable to that of the other models containing more variables. A regression model containing nine independent variables has a good low C_p comparable to that of the other models containing more variables. With respect to the R^2 and C_p values in the graph, the two regression equations containing all variables significant at the .05 level are shown below.

The breaking-strength regression equation with the good high R^2:

$$Y = 4.78 - 0.90X_6 - 0.08X_1X_5 + 2.98\ X_4X_6$$

The breaking-strength regression equation with the low C_p:

$$Y = 6.48 - 0.14X_2 - 1.06X_5 - 1.00X_6 - 0.30X_1X_4 + 0.24X_1X_6 + 0.01X_2X_3 + 0.04X_2X_5 + 0.58X_4X_5 + 2.42X_4X_6$$

where Y = a breaking strength either for the warp or the weft direction

X_1 = BTCA concentration

Table 17 Regression results with the full model for the breaking strength (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	6.21	1.50	0.0001*
BTCA concentration	0.10	0.39	0.8042
IA concentration	-0.20	0.12	0.1089
AA concentration	0.08	0.23	0.7188
monomers/catalyst mole ratio	1.32	1.46	0.3658
curing time	-1.37	0.28	0.0001*
warp (vs. weft)	-0.98	0.44	0.0260*
Interaction Terms			
BTCA*IA	0.03	0.02	0.1720
BTCA*AA	-0.08	0.04	0.0379*
BTCA*monomers/catalyst mole ratio	-0.71	0.35	0.0432*
BTCA*curing time	0.11	0.05	0.0145*
BTCA*warp	0.23	0.07	0.0012*
IA*AA	0.01	0.01	0.3398
IA*monomers/catalyst mole ratio	-0.03	0.11	0.7572
IA*curing time	0.04	0.01	0.0054*
IA*warp	-0.01	0.02	0.7985
AA*monomers/catalyst mole ratio	0.01	0.20	0.9507
AA*curing time	0.02	0.03	0.4216
AA*warp	0.05	0.04	0.1947
monomers/catalyst mole ratio*curing time	0.56	0.23	0.0169*
monomers/catalyst mole ratio*warp	2.41	0.36	0.0001*
curing time*warp	-0.04	0.05	0.4172

Note. The root mean square error of the full model = 0.3565.
$R^2 = 0.8744$
* $p < .05$

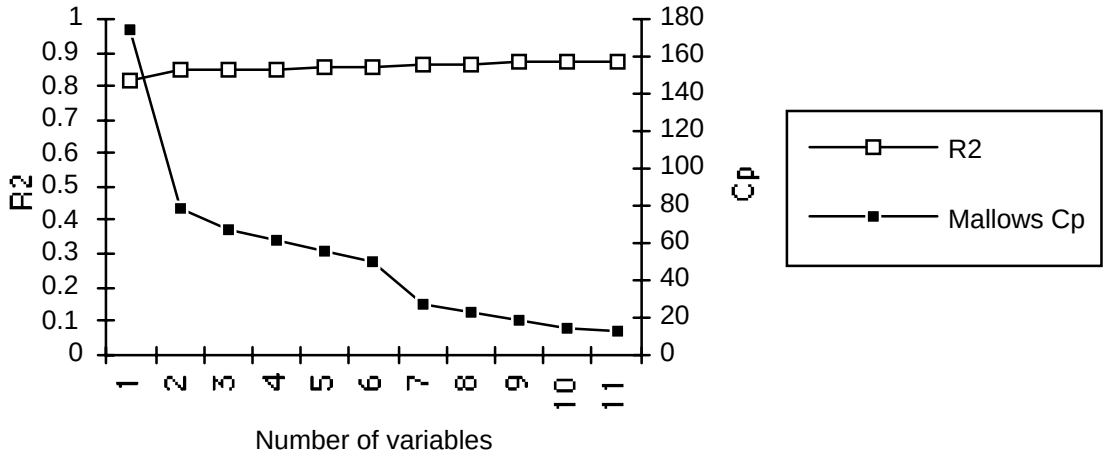


Figure 6. Plot of the values of R^2 and Mallows C_p against the number of variables from the maximum R^2 procedure with the breaking strength data.

X_2 = IA concentration
X_3 = AA concentration
X_4 = mole ratio of monomers to catalyst
X_5 = curing time
X_6 = 1 for warp direction; 0 for weft direction

From the equation with the high R^2, it can be concluded that the fabric direction of the finished specimens significantly affected the breaking strength. According to the high R^2 equation, the warp direction had a higher breaking strength value than the weft direction. Increasing the BTCA concentration and curing time decreased the breaking strength property. Increasing the mole ratio of monomers to catalyst improved this property in the warp direction of the finished fabric specimens.

From the equation with the low C_p, it can be concluded that the fabric direction of the finished specimens significantly affected the breaking strength as well. Increasing the IA concentration and curing time decreased this property. How much the IA concentration decreased the property depended on the AA concentration. The AA concentration in the finish formulation made the IA concentration have less effecting in decreasing this property. The effect of curing time on the breaking strength depended on the IA concentration and the mole ratio of monomers to catalyst. Those latter two variables decreased the ability of the curing time to affect this property. BTCA concentration itself did not affect this property significantly, but the interaction of the BTCA concentration with the mole ratio of monomers to catalyst decreased the breaking strength. Increasing the mole ratio of monomers to catalyst improved the breaking strength in the warp direction of the finished fabric specimens, which is the same conclusion as found in the high R^2 equation. According to the simplified models for the breaking strength, it can be concluded that increased curing time and IA concentration decreased the breaking strength property, while increased BTCA concentration decreased the property when it interacted with the mole ratio of monomers to catalyst. Increasing the AA concentration and the mole ratio of monomers to catalyst improved the breaking strength property.

To assess the predictive ability of the three different equations estimated for the breaking strength, the mean values predicted by the equations were compared with the actual mean values obtained from the measurements in the four trials described in the previous chapter. The prediction results of the mean values of the breaking strength property were obtained by inserting the values of the finishing variables actually used in the finish formulation in each of the four trials in the estimated equations. The results of the mean breaking strength for the warp direction of finished fabric specimens from the equations and from the actual measurements appear in the bar graph in Figure 7. The results of the mean breaking strength for the weft direction of finished fabric specimens from the equations and from the actual measurements appear in the bar graph in Figure 8. As seen in comparing the predicted and measured mean values for Trials 1 and 2 in Figure 7, the equations predicted the breaking strength for the warp direction well when all the independent variables used in the test trials were

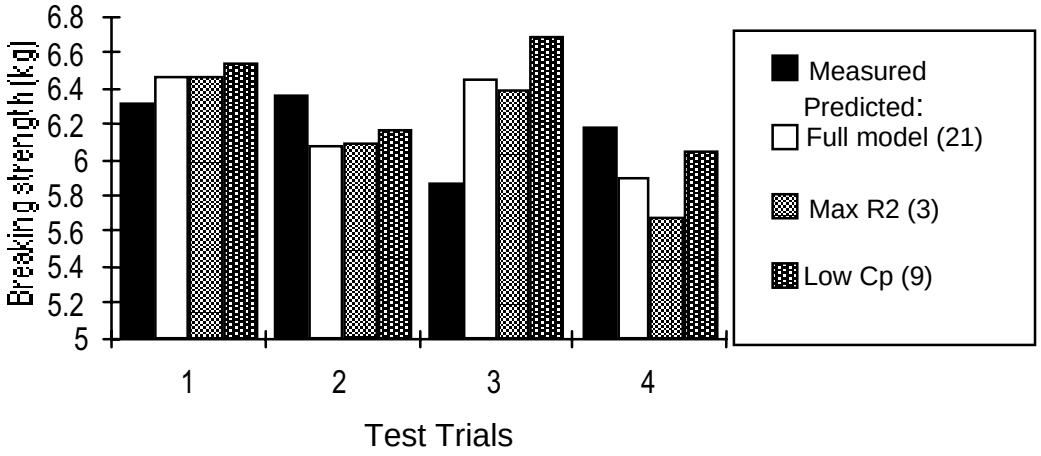


Figure 7. Bar graphs comparing actual mean breaking strength for warp direction obtained from measurements in four test trials with the mean breaking strength predicted by the three different regression equations.

Note. In the legend, each number in parentheses refers to the number of variables contained in the model:

Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.

Test trial 3 had the AA variable outside the original range.

Test trial 4 had the IA and AA variables outside the original range.

in the original ranges, for generating the data for estimating the equations.

The equations did not predict well the breaking strength for the weft direction (see Figure 8) in Trial 1 compared to that in Trial 2 when all the independent variables were in the original ranges. However, the equations did better in predicting the breaking strength when all the independent variables used in the test trials were in the original ranges rather than when some of them were outside the original ranges, as in Trials 3 and 4 in both directions. The full model containing the 21 variables did not predict the property better than the high R^2 model or the low C_p model which contained fewer variables.

Regression Analysis of the Tear Strength

The unit used for the tear strength was the percentage of the total force (3200 g used for testing) that was required to tear the specimens. The data on tear strength used in the regression analysis were not converted to grams (g). The reason to input the percentage of the total force to analyze the regression was that it was obtained directly from the measurement. The percentage of the total force can be converted to tear strength in grams of the specimens by multiplying it by 32.

The full model with the tear strength as the dependent variable was run through the regression procedure. The results for the root mean square error and R^2 of the full model and the significance levels of all variables are reported in Table 18. Referring to p-value in Table 18 with the results of the full model, it can be concluded that three main variables, BTCA concentration, IA concentration, and mole ratio of monomers to catalyst, affected the tear strength property significantly. How these three variables affected this property depended on two interaction terms: BTCA concentration with mole ratio of monomers to catalyst, and IA concentration with mole ratio of monomers to catalyst. These interaction terms affected this property significantly also. The tear strength was not significantly affected by direction of specimens, whether warp or weft. The tear strength results for the warp or weft direction may not be significantly different because of the significant interactions with direction of three other variables; BTCA concentration, AA concentration, and curing time. The full model was difficult to use for interpreting what variables or their interactions very clearly affected this property, so the full model was simplified by running it through the backward elimination procedure to obtain a simpler model.

The model obtained from the backward elimination procedure, with the goal of maximizing the R^2, for this property is reported in Appendix N. Figure 9 shows a graph of the R^2 and Mallows C_p against the number of variables in the stepwise models. This figure shows that a regression model containing just five independent variables has a good high R^2 comparable to that of the other models containing more variables. A regression model containing seven independent variables has a good low C_p comparable to that of the other models containing more variables. With respect to the R^2 and C_p values graph, the two regression equations containing all variables significant at the .05 level are shown below.

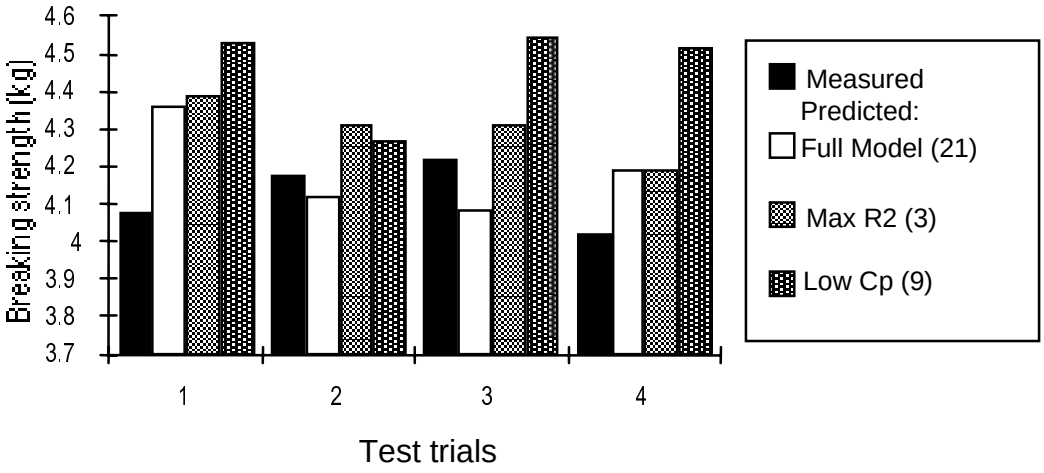


Figure 8. Bar graphs comparing actual mean breaking strength for weft direction obtained from measurements in four test trials with the mean breaking strength predicted by the three different regression equations.

Note. In the legend, each number in parentheses refers to the number of variables contained in the model:

Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.

Test trial 3 had the AA variable outside the original range.

Test trial 4 had the IA and AA variables outside the original range.

Table 18 Regression results with the full model for the tear strength (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	69.23	9.09	0.0001*
BTCA concentration	-8.08	2.37	0.0008*
IA concentration	-2.43	0.74	0.0012*
AA concentration	-0.36	1.42	0.7992
monomers/catalyst mole ratio	-19.33	8.83	0.0296*
curing time	-0.36	1.68	0.8281
warp (vs. weft)	3.76	2.58	0.1475
Interaction Terms			
BTCA*IA	0.18	0.13	0.1841
BTCA*AA	0.17	0.24	0.4827
BTCA*monomers/catalyst mole ratio	5.47	2.11	0.0102*
BTCA*curing time	-0.16	0.28	0.5794
BTCA*warp	-1.16	0.42	0.0067*
IA*AA	0.05	0.07	0.5061
IA*monomers/catalyst mole ratio	1.59	0.66	0.0169*
IA*curing time	0.06	0.09	0.5061
IA*warp	-0.17	0.13	0.1966
AA*monomers/catalyst mole ratio	0.62	1.19	0.6049
AA*curing time	-0.18	0.16	0.2525
AA*warp	0.83	0.24	0.0006*
monomers/catalyst mole ratio*curing time	-2.19	1.41	0.1215
monomers/catalyst mole ratio*warp	1.33	2.11	0.5300
curing time*warp	-1.46	0.28	0.0001*

Note. The root mean square error of the full model = 1.6891.
$R^2 = 0.7963$
* $p < .05$

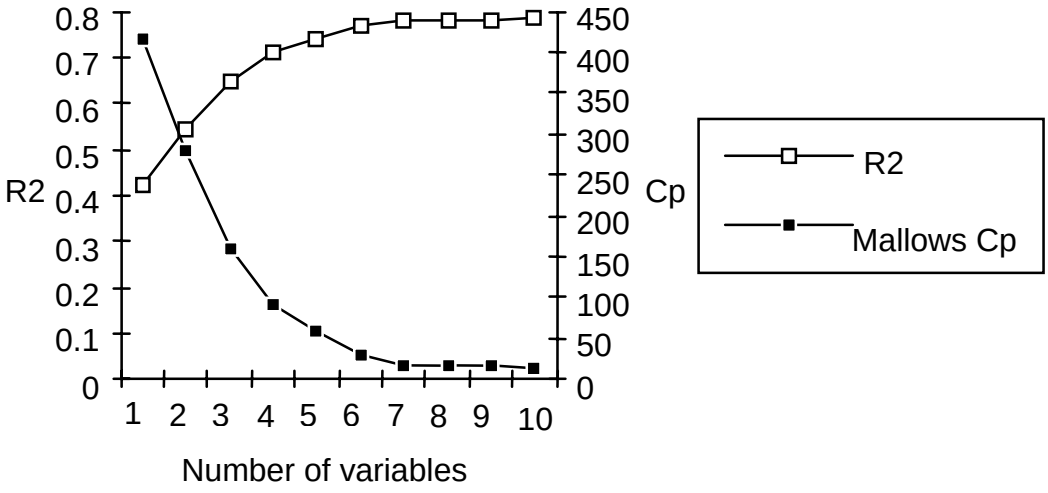


Figure 9. Plot the values of R^2 and Mallow C_p against the number of variables from the maximum R^2 procedure with the tear strength data.

The tear-strength regression equation with the high R^2:

$Y = 52.20 - 7.99X_1 - 0.38X_2 + 0.40X_1X_3 + 5.23X_1X_4 - 3.83\ X_4X_5$

The tear-strength regression equation with the good low C_p:

$Y = 50.94 - 6.94X_1 - 0.38X_2 + 0.21X_1X_3 + 0.46\ X_1X_4 + 0.96X_3X_6 - 3.11X_4X_5 - 1.32X_5X_6$

where Y = a tear strength for either the warp or the weft direction

X_1 = BTCA concentration

X_2 = IA concentration

X_3 = AA concentration

X_4 = mole ratio of monomers to catalyst

X_5 = curing time

X_6 = 1 for warp direction; 0 for weft direction

From the equation with the high R^2, it can be concluded that fabric direction did not significantly affect the tear strength. Increasing the BTCA concentration and the IA concentration decreased the tear strength. From the interaction term of BTCA concentration with AA concentration or with mole ratio of monomers to catalyst, it can be concluded that AA concentration and mole ratio of monomers to catalyst reduced the ability of BTCA to deteriorate the tear strength of the finished fabric specimens. From the interaction term of mole ratio of monomers to catalyst with curing time, it can be concluded that the curing time reduced the ability of the mole ratio of monomers to catalyst to improve the tear strength or in the opposite way, the mole ratio of monomers to catalyst reduced the ability of the curing time to improve the tear strength.

The equation with the low C_p had all the variables contained in the high R^2 equation. In addition, it contained two more interaction terms: AA concentration with the warp direction, and the curing time with the warp direction. It can be concluded that the warp direction of the specimens had a smaller tear strength than the weft direction. The AA concentration improves the property, while the curing time decreases the property for the warp direction. According to the simplified models, it can be concluded that BTCA concentration, IA concentration, and curing time affected negatively the tear strength property, while AA concentration and mole ratio of monomers to catalyst improved the property. The warp and the weft directions of the finished fabric specimens did not have the same tear-strength values, but the values were not significantly different from each other.

To assess the predictive ability of the three different equations estimated for the tear strength, the mean values predicted by the equations were compared with the actual mean values obtained from the measurements in the four trials described previously. The results of the mean tear strength for the warp direction of finished fabric specimens from the equations and from the actual measurements appear in the bar graph in Figure 10. The results of the mean tear strength for the weft direction of finished fabric specimens from the equations and from the actual measurements appear in the bar graph in Figure 11. As seen in comparing the predicted and measured mean values for Trials 1 and 2 in Figures 10 and 11, the equations predicted well the tear strength in both

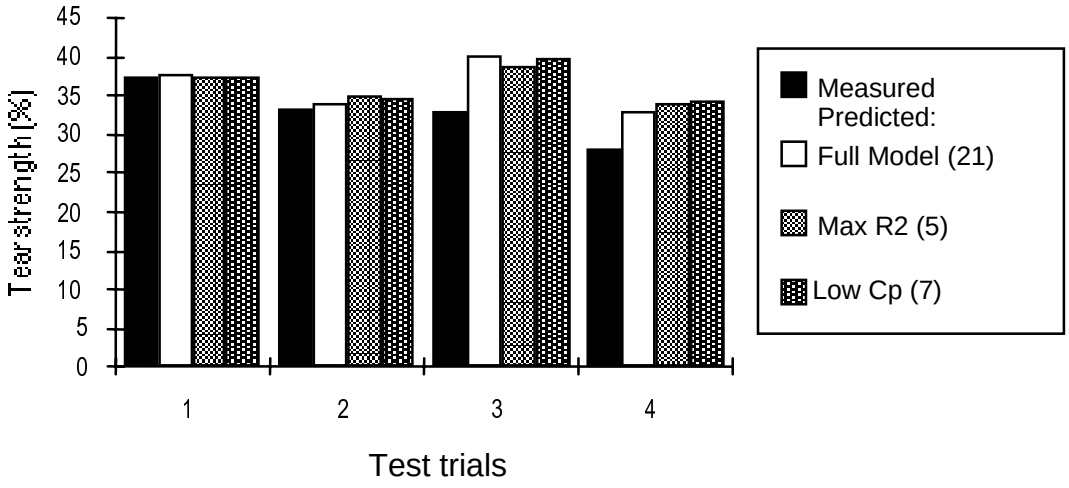


Figure 10. Bar graphs comparing actual mean tear strength for the warp direction obtained from measurements in four test trials with the mean tear strength predicted by the three different regression equations.

Note. In the legend, each number in parentheses refers to the number of variables contained in the model:

Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.

Test trial 3 had the AA variable outside the original range.

Test trial 4 had the IA and AA variables outside the original range.

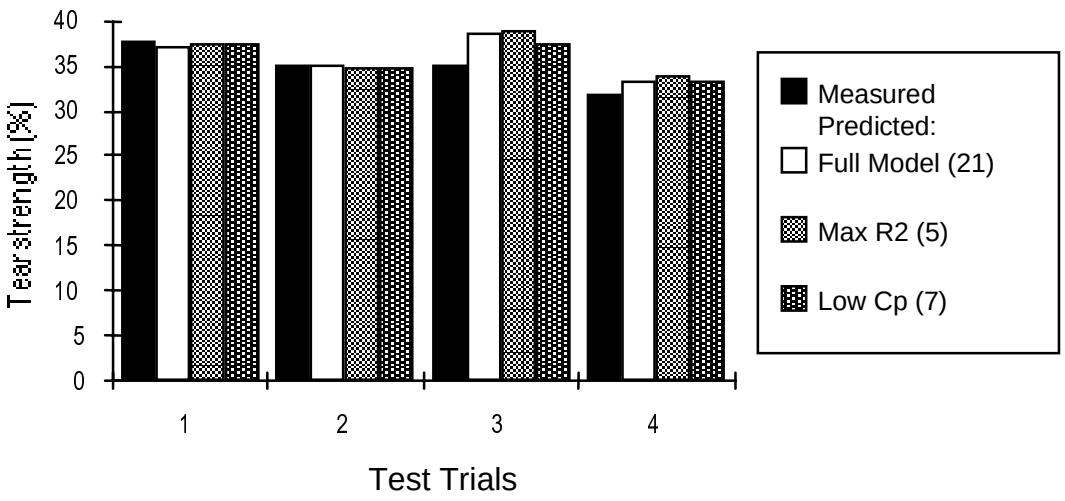


Figure 11. Bar graphs comparing actual mean tear strength for the weft direction obtained from measurements in four test trials with the mean tear strength predicted by the three different regression equations.

Note. In the legend, each number in parentheses refers to the number of variables contained in the model:

Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.

Test trial 3 had the AA variable outside the original range.

Test trial 4 had the IA and AA variables outside the original range.

the warp and in the weft directions when all the independent variables used in the test trials were in the original ranges (as seen in Trials 1 and 2). The equations predicted the tear strength for the weft direction (Figure 11) well in Trial 4, but the predictions were not as accurate for the tear strength in the weft direction in Trial 3 and for the tear strength in the warp direction in Trials 3 and 4. Overall, the equations better predicted the results in the tear strength property when all the independent variables used in the test trials were in the original ranges than when some of them were outside the original ranges. The full model containing the 21 variables did not predict the tear strength better than the high R^2 model or the low C_p model, which each contained fewer variables in their models.

Regression Analysis of the Wrinkle Recovery Angle

The full model with the wrinkle recovery angle as the dependent variable was run through the regression procedure. The results for the root mean square error and R^2 of the full model and the significance levels of all variables are reported in Table 19. According to the regression results for the p-values in Table 19, it can be concluded that the main independent variables did not affect the wrinkle recovery angle of the finished fabric specimens significantly, but some of their interaction terms did. The interaction terms of BTCA concentration with IA concentration and BTCA concentration with mole ratio of monomers to catalyst affected the property significantly. It was hard to determine how these two interaction terms affected the property because their parameter estimates depended on other variables also. The direction of specimens taken from the warp or the weft directions and the crease of specimens folded from face to face or back to back affected the property significantly. The finished specimens taken from the warp face to face had the lowest wrinkle recovery angles compared to those obtained from other directions. The AA concentration, mole ratio of monomers to catalyst, and curing time were three independent variables significantly affecting the wrinkle recovery angle for warp face to face of finished specimens. The mole ratio of monomers to catalyst and curing time affected this property in the warp face to face in a trend of increasing, while the AA concentration affected it in a trend of decreasing (the negative sign of their parameter estimates). The full model was simplified by running it through the backward elimination procedure for obtaining a simpler model. The model obtained from the backward elimination procedure is reported in Appendix O. Figure 12 shows a graph of the R^2 and Mallows C_p against the number of variables in the stepwise models.

Figure 12 shows that a regression model containing four independent variables has a good high R^2 comparable to that of the other models containing more variables. A regression model containing ten independent variables has a good low C_p comparable to that of the other models containing more variables. With respect to the R^2 and C_p values in the graph, the two regression equations containing all variables significant at the .05 level are shown below.

The wrinkle-recovery-angle regression equation with the high R^2:

$$Y= 125.33 + 2.77X_5 -20.87X_6 -9.83X_7 + 0.56X_1X_2$$

Table 19 Regression results with the full model for the wrinkle recovery angle (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	149.65	63.63	0.0192*
BTCA concentration	-2.17	7.41	0.7692
IA concentration	0.94	0.97	0.3321
AA concentration	-0.83	1.86	0.6565
monomers/catalyst mole ratio	-38.87	68.25	0.5693
curing time	1.00	5.21	0.8483
warp face to face	-29.52	4.73	0.0001*
weft back to back	-9.08	4.73	0.0558
weft face to face	-1.75	4.65	0.7067
Interaction Terms			
BTCA*IA	-0.45	0.17	0.0088*
BTCA*AA	0.26	0.31	0.3945
BTCA*monomers/catalyst mole ratio	10.26	2.75	0.0002*
BTCA*curing time	0.35	0.37	0.3346
BTCA*warp face to face	0.50	0.78	0.5207
BTCA*weft back to back	-0.29	0.78	0.7078
BTCA*weft face to face	0.10	0.78	0.8935
IA*AA	0.13	0.08	0.1795
IA*monomers/catalyst mole ratio	1.61	0.86	0.0616
IA*curing time	0.06	0.26	0.8239
IA*warp face to face	-0.09	0.24	0.7078
IA*weft back to back	-0.22	0.24	0.3630
IA*weft face to face	-0.44	0.24	0.0735
AA*monomers/catalyst mole ratio	-2.38	1.55	0.1258
AA*curing time	0.51	0.30	0.0858
AA*warp face to face	-1.04	0.44	0.0189*
AA*weft back to back	-0.21	0.44	0.6299
AA*weft face to face	0.13	0.44	0.7684
monomers/catalyst mole ratio*curing time	-1.77	1.83	0.3346
monomers/catalyst mole ratio*warp face to face	8.12	3.89	0.0374*
monomers/catalyst mole ratio*weft back to back	2.29	3.89	0.5560
monomers/catalyst	5.52	3.89	0.1565

mole ratio*weft face to face			
curing time*warp face to face	1.65	0.47	0.0005*
curing time*weft back to back	0.18	0.47	0.7054
curing time*weft face to face	-0.01	0.12	0.9283

Note. The root mean square error of the full model = 2.6938.
R^2 = 0.9320
* p < .05

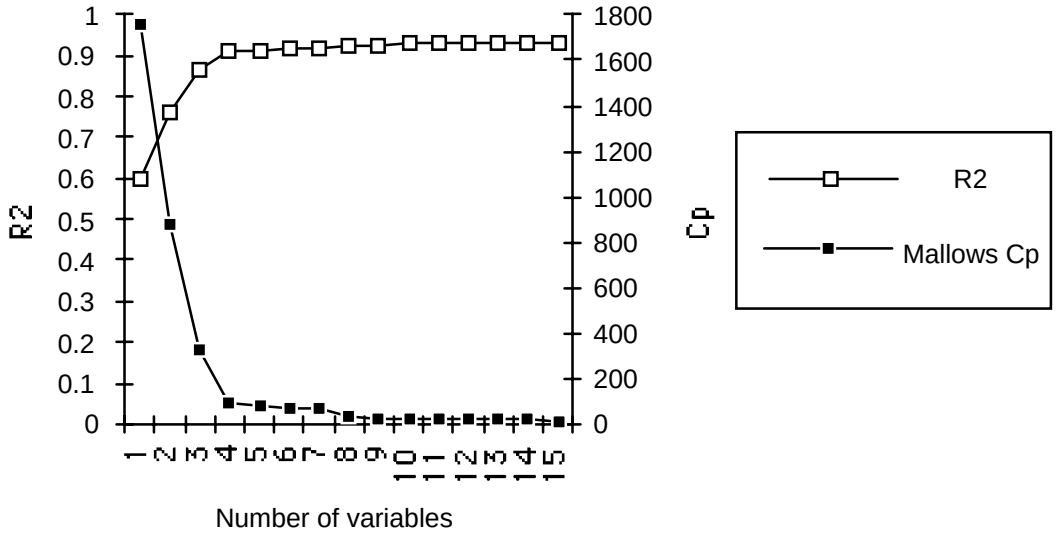


Figure 12. Plot of the values of R^2 and Mallows C_p against the number of variables from the maximum R^2 procedure with the wrinkle recovery angle data.

The wrinkle-recovery-angle regression equation with the low C_p:

$Y = 146.03 - 37.40X_4 - 22.10X_6 - 9.83X_7 - 0.39X_1X_2 + 8.40X_1X_4 + 2.74X_2X_4 - 2.32X_3X_4 + 0.84X_3X_5 - 1.01X_3X_6 + 1.73X_5X_6$

where Y = a wrinkle recovery angle

X_1 = BTCA concentration

X_2 = IA concentration

X_3 = AA concentration

X_4 = mole ratio of monomers to catalyst

X_5 = curing time

X_6 and X_7 = 0 and 0 for warp back to back and for weft face to face

X_6 and X_7 = 1 and 0 for warp face to face

X_6 and X_7 = 0 and 1 for weft back to back

According to the regression model for high R^2, it can be concluded that the curing time significantly affected in a trend of increasing the wrinkle recovery angles of the finished specimens. The interaction term of BTCA concentration with IA concentration affected in a trend of increasing this property also. The specimens taken from the warp face to face had significantly lower wrinkle recovery angles compared to those taken from other directions. The specimens taken from the weft back to back had lower wrinkle recovery angles than those obtained from warp back to back or from weft face to face. The regression model for low C_p contained the independent variables of the mole ratio of monomers to catalyst and its interaction terms with BTCA concentration, with IA concentration, and with AA concentration. Those terms did not appear in the regression model for high R^2. The mole ratio of monomers to catalyst itself affected this property in a trend of decreasing. How much it affected this property depended on the independent variables of BTCA, IA, and AA concentrations. BTCA concentration and IA concentration decreased the ability of mole ratio of monomers to catalyst to deteriorate the property, while AA concentration enhanced the ability of mole ratio of monomers to catalyst to deteriorate the property. Based on the regression equation with high R^2 and some parts of the regression equation with low C_p, it can be concluded that BTCA concentration, IA concentration, and curing time improved the wrinkle recovery angles, while AA concentration and mole ratio of monomers to catalyst decreased the property of the finished fabric specimens.

To assess the predictive ability of the three different regression equations estimated for the wrinkle recovery angles, the mean values of the sum of the average of the property in the warp and weft directions obtained by the equations were compared with the actual mean values of the sum of the average of the property in both directions obtained from the measurements in the test trials. The results of the wrinkle recovery angles from the equations and from the actual measurements appear in the bar graph in Figure 13.

As seen in comparing the predicted and measured values for Trials 1 and 2, the equations predicted the wrinkle recovery angles well when all the independent variables used in the test trials were in the original range used for generating the data for estimating any of the equations. The full model with the

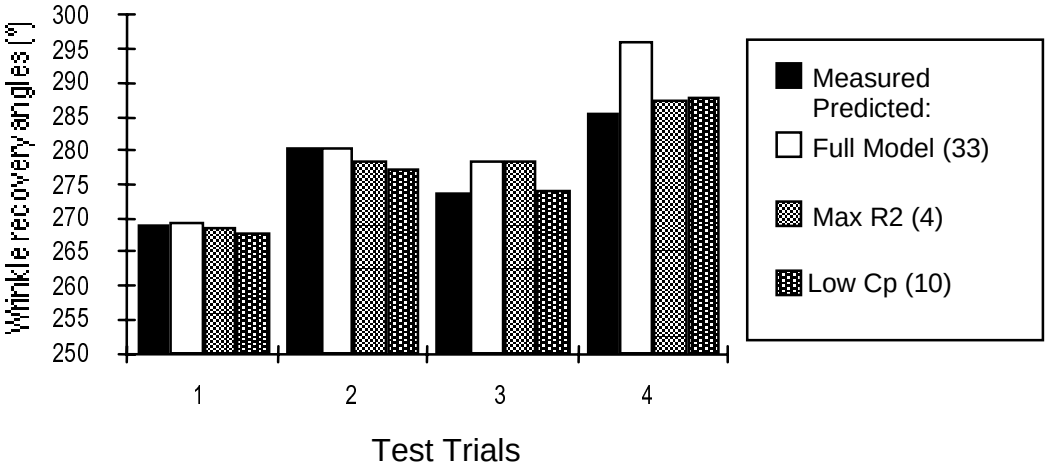


Figure 13. Bar graphs comparing actual mean of the sum of the average of the wrinkle recovery angles in both directions obtained from measurements in four test trials with those predicted by the three different regression equations.
Note. In the legend, each number in parentheses refers to the number of variables contained in the model:
Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.
Test trial 3 had the AA variable outside the original range.
Test trial 4 had the IA and AA variables outside the original range.

larger number of variables did not predict the wrinkle recovery angle results better than the smaller models. When the levels of some variables used in the finish formulation were outside the original range, as in Trials 3 and 4, the prediction was not as accurate.

Regression Analysis of Whiteness Index

The full model with the whiteness index as the dependent variable was run through the regression procedure. The results for root mean square error and R^2 of the full model and the significance levels of all variables are reported in Table 20. By referring to the p-values and parameter estimates of variables in the full model in Table 20, it can be concluded that the BTCA concentration significantly negative affected the whiteness index, while the AA concentration significantly positive affected the whiteness index of the finished fabric specimens. The way the BTCA concentration affected this property depended on the AA concentration and the mole ratio of monomers to catalyst. The AA concentration and the mole ratio of monomers to catalyst decreased the ability of BTCA to deteriorate the whiteness index. The AA concentration improved the property, but its ability depended on the curing time variable. Increasing curing time decreased the ability of the AA concentration to improve the whiteness index of the finished fabric specimens. Increasing curing time not only decreased the ability of the AA concentration but also decreased the ability of mole ratio of monomers to catalyst to improve the property. The full model was simplified by running it through the backward elimination procedure. The model obtained from the backward elimination procedure is reported in Appendix P.

Figure 14 shows a graph of the R^2 and Mallows C_p against the number of variables in the stepwise models. Figure 14 shows that a regression model containing five independent variables had a good high R^2 and a good low C_p comparable to those of the other models containing more variables.

The whiteness-index regression equation with both the high R^2 and the low C_p:

$$Y= 75.31 - 13.24X_1 - 0.58X_2 + 0.96X_1X_3 + 9.82X_1X_4 - 2.21X_4X_5$$

where Y = a whiteness index

X_1 = BTCA concentration

X_2 = IA concentration

X_3 = AA concentration

X_4 = mole ratio of monomers to catalyst

X_5 = curing time

All the independent variables contained in the above regression equation were significant at the .05 level. From that equation, it can be concluded that BTCA concentration and IA concentration affected the whiteness index of the finished fabric specimens significantly in a trend of decreasing. The AA concentration and mole ratio of monomers to catalyst reduced the ability of the BTCA concentration to deteriorate this property. An increased curing time decreased the ability of mole ratio of monomers to catalyst to improve the whiteness index of the finished specimens. Based on the above regression equation, BTCA concentration, IA concentration and curing time tended to decrease the whiteness index, while AA concentration and mole ratio of monomers to catalyst tended to improve the property. To assess the

Table 20 Regression results with the full model for the whiteness index (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	60.77	10.86	0.0001*
BTCA concentration	-13.98	2.85	0.0001*
IA concentration	-0.86	0.89	0.3331
AA concentration	4.00	1.71	0.0205*
monomers/catalyst mole ratio	16.07	10.59	0.1309
curing time	3.49	2.01	0.0855
Interaction Terms			
BTCA*IA	0.28	0.16	0.0849
BTCA*AA	0.59	0.29	0.0408*
BTCA*monomers/catalyst mole ratio	7.94	2.55	0.0021*
BTCA*curing time	0.53	0.34	0.1197
IA*AA	0.02	0.09	0.8368
IA*monomers/catalyst mole ratio	-0.08	0.80	0.9197
IA*curing time	-0.17	0.11	0.1137
AA*monomers/catalyst mole ratio	-2.13	1.44	0.1400
AA*curing time	-0.56	0.19	0.0038*
monomers/catalyst mole ratio*curing time	-4.39	1.70	0.0106*

Note. The root mean square error of the full model = 1.7665.
$R^2 = 0.8210$
* $p < .05$

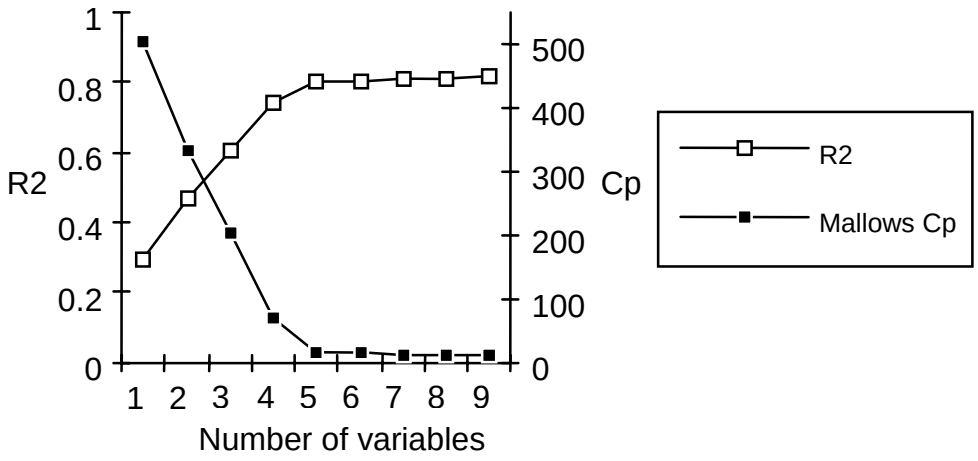


Figure 14. Plot of the R^2 and Mallow C_p against the number of variables from the maximum R^2 procedure with the whiteness index data.

predictive ability of the three different regression equations estimated for the whiteness index, the mean values of the property determined by the equations were compared with the actual mean values of the property obtained from the measurements in the test trials. The results the whiteness index determined from the equations and from the actual measurements appear in the bar graph in Figure 15.

As seen in comparing the predicted and measured mean values for the four trials in Figure 15, the equations predicted the whiteness index well when all the independent variables used in Test trial 1 were in the original ranges for generating the data for estimating the equations. The equations predicted the whiteness index in Trial 2 as well as that in Trials 3 and 4. Overall, the equations better predicted the results in the whiteness index property when all the independent variables used in the test trials were in the original ranges than when some of them were outside the original ranges, as in Trials 3 and 4. The full model containing the fifteen variables did not predict the whiteness index better than the high R^2 model or the low C_p model, which each contained fewer variables in their model.

Regression Analysis of Durable Press Rating

The full model with the durable press rating as the dependent variable was run through the regression procedure. The results for the root mean square error and the R^2 of the full model and the significance levels of all variables are reported in Table 21. By referring to the p-values and parameter estimates of variables in the full model in Table 21, it can be concluded that the observers did not significantly affect the durable press property. Therefore, the observer factor was taken out of the full model. A reason to remove the observer factor is because the number of regression equations can be reduced from three equations (each equation for each observer grading the property) to one equation for the overall durable press rating. The full model without the observer factor was used as a full model for this property and re-run through the regression procedure. The results for the root mean square error and the R^2 of the full model without the observer factor and the significance levels of all variables are reported in Table 22.

From the regression results of the p-values and parameter estimates of variables in Table 22, it can be concluded that the four main independent variables of IA concentration, AA concentration, mole ratio of monomers to catalyst, and curing time significantly affected the durable press rating. The number of launderings significantly affected the property also. The BTCA concentration by itself did not significantly affect this property, but its interaction terms with the number of washings significantly affected this property, which implies that the BTCA concentration tended to decrease the durable press ratings after the fifth launderings of the finished fabric specimens. IA concentration, AA concentration, and mole ratio of monomers to catalyst were other variables that tended to decrease the durable press ratings after the fifth launderings of the finished fabric specimens. IA concentration, AA concentration, and curing time affected the ability of mole ratio of monomers to catalyst to improve the durable press rating. It was difficult to determine what role each independent variable played in affecting the durable press rating because there were many interaction terms influencing each of the main

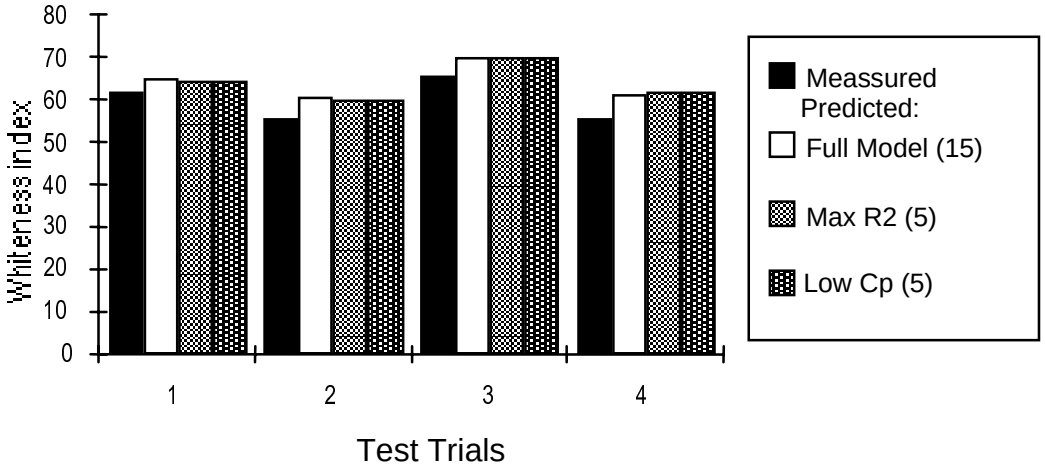


Figure 15. Bar graphs comparing actual mean whiteness index obtained from measurements in four test trials with mean whiteness index predicted by the three regression equations.

Note. In the legend, each number in parentheses refers to the number of variables contained in the model:

Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.

Test trial 3 had the AA variable outside the original range.

Test trial 4 had the IA and AA variables outside the original range.

Table 21 Regression results with the full model for the durable press rating (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	-2.29	0.89	0.0001*
BTCA concentration	0.27	0.23	0.2305
IA concentration	0.38	0.07	0.0001*
AA concentration	0.49	0.14	0.0004*
monomers/catalyst mole ratio	4.34	0.86	0.0001*
curing time	0.88	0.16	0.0001*
fifth washings	1.53	0.25	0.0001*
2nd observer	0.25	0.30	0.4063
3rd observer	-0.10	0.30	0.7644
Interaction Terms			
BTCA*IA	0.00	0.01	0.8636
BTCA*AA	-0.03	0.02	0.2293
BTCA*monomers/catalyst mole ratio	0.24	0.20	0.2293
BTCA*curing time	-0.05	0.03	0.0591
BTCA*fifth washings	-0.18	0.04	0.0001*
BTCA*2nd observer	-0.07	0.05	0.1717
BTCA*3rd observer	-0.10	0.05	0.0460*
IA*AA	-0.01	0.01	0.0591
IA*monomers/catalyst mole ratio	-0.36	0.06	0.0001*
IA*curing time	0.00	0.01	0.8636
IA*fifth washings	-0.09	0.01	0.0001*
IA*2nd observer	0.00	0.02	0.9162
IA*3rd observer	0.01	0.02	0.4615
AA*monomers/catalyst mole ratio	-0.33	0.11	0.0036*
AA*curing time	-0.01	0.01	0.6062
AA*fifth washings	-0.07	0.02	0.0021*
AA*2nd observer	0.00	0.03	0.9162
AA*3rd observer	-0.02	0.03	0.4615
monomers/catalyst mole ratio*curing time	-0.72	0.13	0.0001*
monomers/catalyst mole ratio*fifth washings	-0.83	0.20	0.0001*
monomers/catalyst mole ratio*2nd observer	-0.23	0.25	0.3438
monomers/catalyst mole ratio*3rd observer	0.29	0.25	0.2474
curing time*fifth washings	0.00	0.03	1.0000

curing time*2nd observer	0.03	0.03	0.3438
curing time*3rd observer	0.01	0.03	0.7523

Note. The root mean square error of the full model = 0.2424.
R^2 = 0.6634
* $p < .05$

Table 22 Regression results with the full model not containing the observer factor for the durable press rating (1 degree of freedom for every variable)

Variable	parameter Estimates	Standard Error	p-value
Intercept	-2.23	0.87	0.0104*
BTCA concentration	0.22	0.23	0.3362
IA concentration	0.39	0.07	0.0001*
AA concentration	0.48	0.14	0.0005*
monomers/catalyst mole ratio	4.36	0.85	0.0001*
curing time	0.90	0.16	0.0001*
fifth washings	1.56	0.25	0.0001*
Interaction Terms			
BTCA*IA	0.00	0.01	0.8639
BTCA*AA	-0.03	0.02	0.2304
BTCA*monomers/catalyst mole ratio	0.24	0.20	0.2304
BTCA*curing time	-0.05	0.03	0.0597
BTCA*fifth washings	-0.18	0.04	0.0001*
IA*AA	-0.01	0.01	0.0597
IA*monomers/catalyst mole ratio	-0.36	0.06	0.0001*
IA*curing time	0.00	0.01	0.8639
IA*fifth washings	-0.09	0.01	0.0001*
AA*monomers/catalyst mole ratio	-0.33	0.11	0.0037*
AA*curing time	0.00	0.01	0.6071
AA*fifth washings	-0.07	0.02	0.0021*
monomers/catalyst mole ratio*curing time	-0.72	0.13	0.0001*
monomers/catalyst mole ratio*fifth washings	-0.83	0.20	0.0001*
curing time*fifth washings	0.00	0.03	1.0000

Note. The root mean square error of the full model = 0.2429.
$R^2 = 0.6530$
* $p < .05$

independent variables. The full model without the observer factor was simplified by running it through the backward elimination procedure. The model obtained from this procedure is reported in Appendix Q. A bar graph of the number of variables against the R^2 and Mallows C_p is shown in Figure 16. Because of the data reported in Figure 16, a regression model containing five independent variables was chosen as a high R^2 due to its R^2 being comparable to that of other models containing more variables. A regression model containing eleven independent variables was chosen as a low C_p due to its C_p being as low as that of other models containing more than eleven variables.

The durable-press-rating regression equation with the high R^2:

$$Y= 2.90 + 0.60X_5 + 0.16X_1X_4 - 0.05X_2X_6 - 0.06X_3X_6 - 0.56X_4X_5$$

The durable-press-rating regression equation with the low C_p:

$$Y= -0.57 + 0.34X_2 + 3.43X_4 + 0.74X_5 + 1.59X_6 + 0.26X_1X_4 - 0.18X_1X_6 - 0.36X_2X_4 - 0.09X_2X_6 - 0.08X_3X_6 - 0.72X_4X_5 - 0.83X_4X_6$$

where Y = a durable press rating for either after one or five launderings

X_1 = BTCA concentration

X_2 = IA concentration

X_3 = AA concentration

X_4 = mole ratio of monomers to catalyst

X_5 = curing time

X_6 = 0 for first laundering, 1 for fifth laundering

From the equation for high R^2, it can be concluded that the main effect of curing time was a significant improvement in the durable press rating property. The interaction term of BTCA concentration and the mole ratio of monomers to catalyst improved this property. The interaction term of the mole ratio of monomers to catalyst with the curing time decreased the property of the finished fabric specimens. IA and AA concentrations decreased the durable press rating after the fifth launderings of the finished fabric specimens. This may imply that the crosslinking bonds formed by the reaction of IA/AA mixture were not stable in laundering. The additional number of launderings could break the crosslinking bonds and form a new bond that was less effective in the finished fabric specimens after the fifth laundering than that in the finished fabric specimens after one laundering. The regression equation with a high R^2 could indicate that BTCA and curing time improved the durable press rating of finished fabric specimens after the first laundering, while the mole ratio of monomers to catalyst tended to decrease this property. There are no IA and AA concentrations involved in the regression of the durable press rating after one washing. After the fifth launderings, the IA and AA concentrations tended to decrease the property.

From the regression equation with low C_p, it can be concluded that the three main independent variables of IA concentration, mole ratio of monomers to catalyst, and curing time, tended to increase the durable press rating, but their ability to improve depended on their interactions with other variables. The interaction term of mole ratio of monomers to catalyst with BTCA improved the

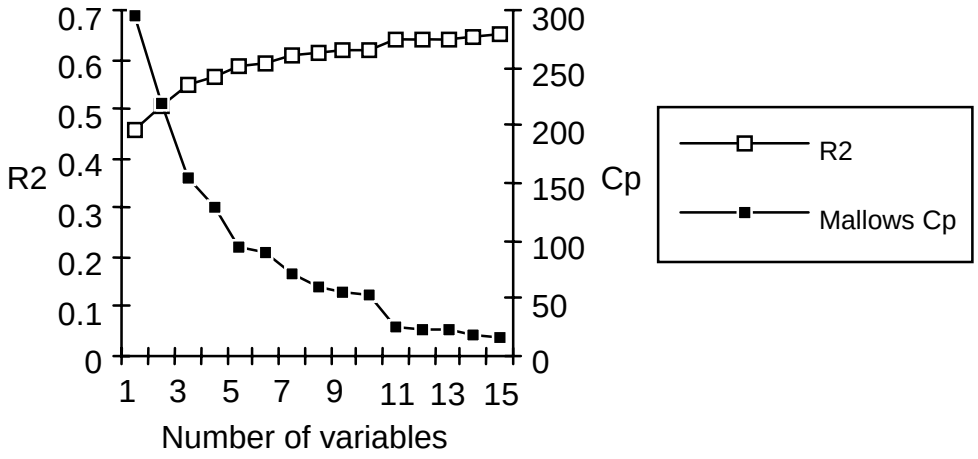


Figure 16. Plot of the values of R^2 and Mallows C_p against the number of variables from the maximum R^2 procedure with the durable press rating data.

property, while the interaction terms of IA concentration with mole ratio of monomers to catalyst and mole ratio of monomers to catalyst with curing time decreased the property. The variables of BTCA, IA, and AA concentrations, and mole ratio of monomers to catalyst decreased the property after the fifth washings of finished fabric specimens. The regression with low C_p could indicate that BTCA, mole ratio of monomers to catalyst, and curing time improved the durable press rating after the first laundering, while the IA concentration decreased the property. The AA concentration was not involved in the equation for the first laundering. After the fifth launderings, IA and AA concentrations decreased the durable press rating of the finished fabric specimens. BTCA concentration, mole ratio of monomers to catalyst, and curing time tended to improve the property, but the improvement of the property after the fifth laundering was less than that of the property after the first laundering. Those two simplified models did not provide the same conclusion on how mole ratio of monomers to catalyst influenced on the property. For the regression with high R^2, the mole ratio of monomers to catalyst tended to decrease the property. For the regression with low C_p, the mole ratio of monomers to catalyst tended to improve the property.

To assess the predictive ability of the three different regression equations estimated for the durable press rating for either after the first washing or the fifth washing, the mean values of the property obtained by the equations were compared with the actual mean values of the property obtained from the measurements in the test trials. The results of the durable press rating after the first washing from the equations and from the actual measurements appear in a bar graph in Figure 17. The results of the durable press rating after the fifth washing from the equations and from the actual measurements appear in a bar graph in Figure 18. As seen in comparing the predicted and measured mean values for the four trials in Figures 17 and 18, the equations predicted better the durable press rating after the first washing of the finished fabric specimens in the first three trials than in Trial 4; the equations predicted the durable press rating after the fifth washings better in Trials 2, 3, and 4 but not in Trial 1.

The prediction of durable press rating by the three different regression equations did not correspond to the previous predictions of other properties. It may be due to the low R^2 of the durable press rating regressions. The R^2 of the durable press rating equation was about 0.6, which was considered a low R^2 compared to that of the other regressions for other properties. The low R^2 implied that the dependent variable cannot be predicted well by the independent variables. Therefore, the prediction of the durable press rating by the regressions could be either very good or bad, by chance.

Summary of the Regression Analysis

The three different regression equations having high R^2 values of about 0.75 and up predicted the values of the fabric properties well compared to the measured values obtained from the measurements in four test trials. The durable press rating regression equation having an R^2 of about 0.6 did not predict the property well compared to that of other regression equations having

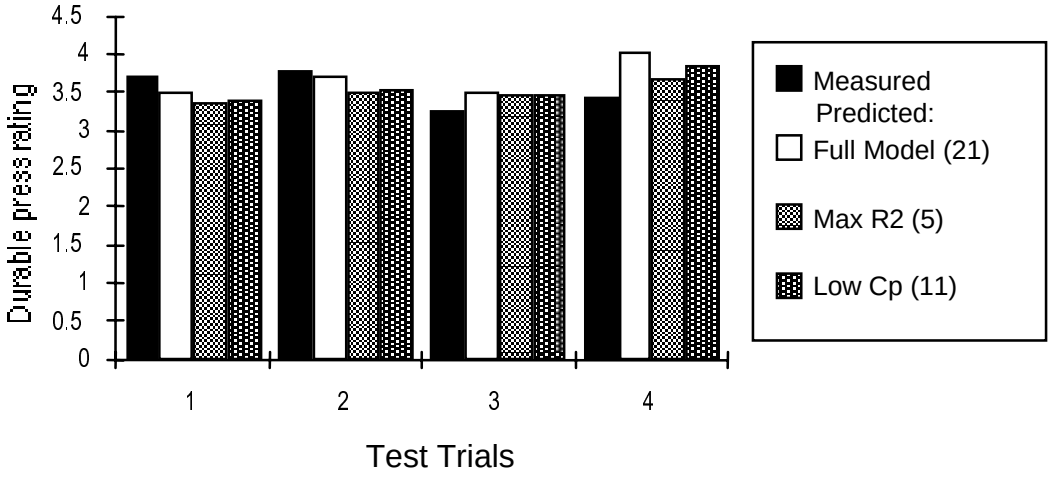


Figure 17. Bar graphs comparing actual mean durable press rating after first washing of finished specimens obtained from measurements in four test trials with mean durable press rating after first washing predicted by the three regression equations.

Note. In the legend, each number in parentheses refers to the number of variables contained in the model:

Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.

Test trial 3 had the AA variable outside the original range.

Test trial 4 had the IA and AA variables outside the original range.

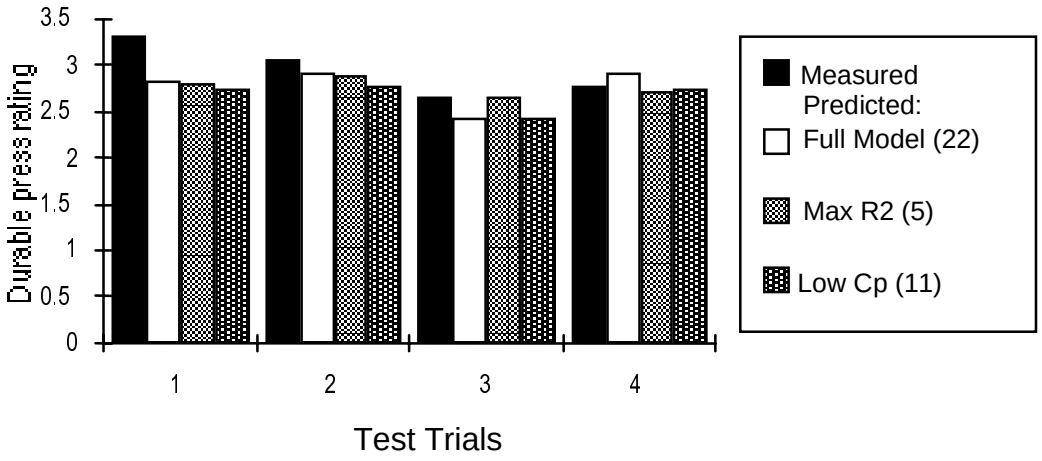


Figure 18. Bar graphs comparing actual mean durable press rating after fifth washing of finished specimens obtained from measurements in four test trials with mean durable press rating after fifth washings predicted by the three regression equations.

Note. In the legend, each number in parentheses refers to the number of variables contained in the model:

Test trials 1 and 2 had all the independent variables in the ranges used to generate the original data with which the regression equation was estimated.

Test trial 3 had the AA variable outside the original range.

Test trial 4 had the IA and AA variables outside the original range.

R^2 larger than 0.75. The regression equation having high R^2 predicted the fabric properties of breaking strength, tear strength, wrinkle recovery angle, and whiteness index well when all the independent variables used in the equation were in the original range, of the data used for estimating the equations. The regression equation having a low R^2 predicted the durable press rating of finished fabric well or not, by chance (as seen in the prediction results of the durable press rating). The simplified regression model containing few variables such as the high R^2 regression equation predicted the result of fabric property as effectively as the more complicated model such as the full model regression equation. The regression analysis showed that some of the interaction terms of each of the main independent variables influenced the main variables affecting the fabric properties. The interaction terms could either enhance or deteriorate the ability of the main variables to affect the fabric properties. The high R^2 equation for each fabric property contained some interaction terms. The research hypotheses 1 to 3 were accepted. Each of the main independent variables was dependent on other variable to affect the fabric properties.

From the simplified regression equations for breaking strength, tear strength, and whiteness index, it can be concluded that the BTCA and IA concentrations and the curing time tended to decrease those three fabric properties, while the AA concentration and the mole ratio of monomers to catalyst tended to improve those properties. These conclusions obtained from the regression analysis were the same as those obtained from examining the mean values of the fabric properties.

From the simplified regression equations for wrinkle recovery angle, it can be concluded that the BTCA and IA concentrations and the curing time tended to improve the wrinkle recovery angles of the finished fabric specimens, while the AA concentration and the mole ratio of monomers to catalyst tended to decrease the angles. The conclusions obtained from the regression analysis were the same as those obtained from examining the mean values of the wrinkle recovery angles of fabric specimens finished with the 32 different BTCA/IA/AA formulations.

For the durable press rating property, the conclusion obtained from the regression analysis was different from that obtained from examining the mean values of this property. According to the regression analysis, the BTCA concentration and the curing time tended to improve the property, while the IA and AA concentrations tended to decrease the property. The mole ratio of monomers to catalyst could affect either the improvement or the deterioration the property depending on the number of launderings, one or five, of the finished fabric specimens. According to the mean values of this property, the BTCA and IA concentrations and the curing time tended to improve the durable press rating of the finished fabric specimens, while the AA concentration and the mole ratio of monomers to catalyst tended to decrease the properties

The possible reasons that support the above conclusions are as follows:

(a) The mechanical properties of the finished fabrics can be ascribed to acid damage and the restriction of stress distribution within the fibers (Choi, 1992; Shin, Hollies & Yen, 1989). The reagents applied on the cotton fabrics were acidic agents

that would reduce the strength of the cotton fabrics. In general, for a given agent, the higher the concentration of reagents in the finish formulation, the larger the amount of the reagents forming the covalent crosslinkages between the reagents and the fibers, and the higher the wrinkle resistance of the finished fabrics. It can be seen in the study that the higher concentration levels of the crosslinker (BTCA) and the IA concentration in the polymer builder (mixture of IA/AA) provided a higher degree of wrinkle recovery angle than did the low concentration levels. The highest degree of wrinkle resistance in the fabrics can reduce the strength of the fabrics too much because of extreme stress restriction in the cellulosic fibers so that they would no longer be useful. Strength loss and wrinkle resistance would be compromised.

(b) The breaking and tear strength of the finished fabrics can be improved by reducing the restriction of stress distribution within the fibers caused by the rigid crosslinking (Bertoniere et al., 1974; Hollies & Getchell, 1967; Rowland et al., 1974; Shin et al., 1989). Breaking strength losses result from decreased flexibility of the structure inside the fibers. Consequently, poor load distribution among the fibers permits the low load to break the fibers when the cotton fibers are crosslinked. Tear strength losses result from poor fiber extendibility that causes decreased mobility of crosslinked structures formed in the cellulosic fibers. Increasing the mole ratio of monomers to catalyst and the concentration of AA may increase the rate of the polymerization of the polymer builder (mixture of IA/AA), so the polymer chain may be longer than that derived from the low level of these two variables. Long chains of the polymer may decrease the stress within the fibers, thus tending to improve flexibility and mobility in the cotton fibers. Increasing those two variables may cause the mechanical properties of the breaking and tear strength to improve because the polymer chains may be longer and more flexible.

(c) If finished fabrics are exposed to high heat for a long time, the heat will scorch the fabrics and degrade polymer chains. Therefore, a long curing time may tend to decrease the breaking and tear strength and also the whiteness index of the finished fabrics.

(d) Yellowing of the finished fabrics may be caused by the number of double bonds of chemical reactants. Itaconic acid (IA) and acrylic acid (AA) were the only unsaturated monomers. According to the discussion of the preliminary results, adding acrylic acid in the finish formulation reduced the observed yellowing of the finished fabrics. Increasing the concentration of AA may increase the rate of polymerization of the polymer builder mixture. The unsaturated monomers may be polymerized by each other or with themselves and form a polymer having fewer unsaturated bonds. Fewer double bonds in the finish formulation applied to the cotton fabrics may cause less yellowing or a higher whiteness index of the finished fabrics.

(e) Networks of crosslinking within cotton fibers will pull the cellulose chains back into position after the removal of a distorting force, so the fabric will resist wrinkling (Andrews, 1992, 1995; Cooke & Weighmann, 1982 a&b; Smith & Block, 1982 a&b). The number of crosslinks formed in the fiber is related to the concentration of the reagents in the finishing bath and to the amount of the reagents bound to the cellulosic fibers. The curing condition and the catalyst are important factors to cause the reactants to form the networks with the cellulose effectively and

maximally. BTCA by itself can react with cellulose and form ester crosslinking networks effectively under heat and catalyst (Andrews, Welch & Trask-Morrell, 1989; Welch, 1990; Welch & Andrews, 1989 a&b, 1990). The different amounts of catalyst used in this study were found to affect the ability of the BTCA/IA/AA reactant combinations to form ester crosslinks with cellulose. High curing temperature and long curing time produce the maximum reaction of the BTCA/IA/AA reactant combinations. Because the curing temperature of 180°C was a constant factor in this study, only the variable curing time could differentially affect the performance of the BTCA/IA/AA reactant combinations in forming ester crosslinks with cellulose. Increasing the concentration of BTCA and the curing time may increase the ester crosslinking networks in cellulose, so that the wrinkle recovery angle property of the finished fabrics may be increased.

(f) Measurement of the durable press rating could be considered a method that simulates a real life situation. Cloth is laundered with some kinds of commercial bleaches and detergents. People expect to see smoothness in the cloth when it comes out from a dryer. According to the method for the durable press rating in this research, the laundered specimens were compared with AATCC 3-D Smoothness Appearance Replicas. Fabrics that have the good crosslink networks are expected to perform well both in the wrinkle recovery angle and the durable press rating. Fabrics having a high degree of wrinkle recovery angle should have a high durable press rating. Therefore, those variables improving the wrinkle recovery angle property should improve the durable press rating as well. However, the crosslinking bonds formed in cellulose should resist bleaches, detergents, and distortion forces in the washing machine, so that the crosslinking bonds can remain in good crosslink networks. In this study, the durable press rating of the fabric finished with the BTCA/IA/AA reactant combinations declined after the fifth launderings. A reason may be that the long chain polymer obtained from the polymerization of BTCA/IA/AA combinations may contain a certain amount of unsaturated side groups (in the mixture of IA/AA). Unsaturated side groups are considered a weak point of a polymer because they are an origin point to cause polymer degradation under circumstances such as laundering conditions.

(g) Increased concentration of IA resulted in an increase of the wrinkle recovery angle. Itaconic acid by itself was not effective enough to be used as a durable press finish like BTCA because it did not have enough carboxylic groups in its molecule. By the polymerization process with potassium persulfate (initiator) and acrylic acid (co-monomer), the IA and AA reactants could be a part of a polymer that has a longer chain compared with those of IA or AA monomers and that has enough carboxylic groups to react with cellulose and form effective crosslink networks within the cellulose.

Comparison of Measured Properties of the Fabric Finished with the BTCA/IA/AA Combinations with Those of Fabric Finished with BTCA only or with DMDHEU

DMDHEU is the most effective durable press finish now in use on cotton fabrics in the textile finishing area, whereas BTCA is the most effective nonformaldehyde durable press finish to be considered for replacing DMDHEU. DMDHEU is a formaldehyde durable press finish that has the disadvantage of the

toxic formaldehyde vapors. BTCA reactant is a nonformaldehyde durable press finish that is not harmful to humans; however, BTCA has a disadvantage in the cost, being more expensive than DMDHEU. BTCA is comparable to DMDHEU in the durable press and mechanical properties of the finished fabrics, as shown below. Below is a summary of the measured properties of the cotton 3/1 twill woven fabric with no finish and when finished with either 6.3% BTCA or 12% DMDHEU. The measured fabric properties of the fabric finished with either BTCA only or DMDHEU are standard properties for comparing with those of fabric finished with other finish formulations of BTCA/IA/AA combinations as described in the previous chapter. The results on those properties of the fabrics with no finish and when finished with BTCA or DMDHEU are reported below as the mean values, each rounded up as was described previously when discussing means and standard deviations. The retentions of breaking and tear strength are reported in parentheses.

	Fabric Finish		
Fabric Property	None	BTCA	DMDHEU
Breaking strength (kg) in warp	9.40	5.98 (64%)	5.76 (61%)
Breaking strength (kg) in weft	7.51	4.18 (56%)	3.99 (53%)
Tear strength (g) in warp	1944	1156 (59%)	952 (49%)
Tear strength (g) in weft	1852	1176 (63%)	1016 (55%)
Wrinkle recovery angle (°)	146°	268°	288°
Whiteness index	81	71	73
1st durable press rating	1.5	3.6	3.8
5th durable press rating	1.3	3.4	3.4

The properties of the fabric finished with either the DMDHEU or only the BTCA reactant showed some significant differences from each other, but no significant differences from others. The wrinkle recovery angles (F (1, 16) = 93.12, p = .0001), the whiteness indexes (F (1, 10) = 19.13, p = .0014), and the tear strengths (F (1, 12) = 4.94, p = .0463) that resulted from using the above two finishing reactants were significantly different at .05 level, but the breaking strengths (F (1, 22) = 2.69, p = .1150) were not significant different. The durable press ratings after the first laundering were significantly different at the .05 level (F (1, 16) = 7.69, p = .0136) for the specimens finished with BTCA vs. DMDHEU, but there was no significant difference in the ratings after the fifth launderings (F (1, 16) = 0.00, p = 1.0). The overall durable press ratings (combining the durable press ratings after the first and fifth launderings) of the finished fabrics using the above two reactants were not significantly different at the .05 level (F (1,32) = 3.70, p = 0.0632). The fabric finished with either of the durable press finishes had better durable press performance (wrinkle recovery angle and durable press rating) than the fabric with no finish, but the mechanical properties (breaking and tear strengths) and the whiteness index of the finished fabric were poorer than those of the fabric with no finish.

This research was designed to evaluate the efficacy of using BTCA in combination with other cheaper reactants (principally the mixture of IA/AA) as an alternative finish formulation to reduce the cost by reducing the amount of BTCA used. An important aspect of evaluating the efficacy of the BTCA/IA/AA combinations as durable press finishes is to compare the resultant fabric properties from those combinations and from BTCA alone or the DMDHEU

reactant. Based on the results of the research, some BTCA/IA/AA combinations provided good results on the wrinkle recovery angle and breaking and tear strength compared to results obtained from using either the BTCA or the DMDHEU reactant. Table 23 is a re-inserted table (same as Table 8) of the 32 different formulations of BTCA/IA/AA. Table 24 contains a summary of the mean results on the measured properties of the fabric finished with each of the 32 finish formulations of the BTCA/IA/AA combinations. The mean values for breaking strength and tear strength are reported as the retentions in Table 24, instead of the units of kilograms and grams force for the breaking and tear strengths, respectively. The raw data of five properties of fabric specimens finished with each of the 32 finish formulations are reported in Appendices R to V. According to the results on the mean values of properties in Table 24, the 2%BTCA/9.6%IA/1.77%AA and 2%BTCA/9.6%IA/3.54%AA finish formulations, each with curing for 90 sec. and with either 1:1 or 1:0.8 mole ratio of monomers to catalyst (Treatments #7, 8, 23 and 24 in3 Table 23) provided wrinkle recovery angles of about 265° to 268°, which are equivalent to the 268° angle of fabric finished with the BTCA reactant. Reducing the concentration of IA to 6.4% o.w.b. in the above formulations also provided a wrinkle recovery angle of the finished fabrics in the same range (Treatments #1, 17, and 18 in Table 23), but the finish formulation had to be cured for 3 min. at 180 °C. When the curing time of the fabric finished with the same above formulations was increased to 3 min. (Treatments #3, 4, 19, and 20 in Table 23), an increase of 8° to 10° wrinkle recovery angles resulted. Increasing the concentration of BTCA to 3% o.w.b. in the above formulations (Treatments # 15, 16, 31, and 32) increased the wrinkle recovery angles about 8° to 10° as well. When the concentration of BTCA in the above finish formulations was increased to 3% o.w.b. and the curing time to 3 min.(Treatments # 11, 12, 27, and 28), good wrinkle recovery angles of the finished fabric, around 280° to 285°, resulted. This was comparable to the wrinkle recovery angles of fabric finished with the DMDHEU reactant.

Most of the 32 finish formulations provided mean breaking and tear strength retentions of the finished fabric comparable to those that resulted with either the BTCA or DMDHEU reactant. Two other measured properties, whiteness index and durable press rating, need to be considered. The BTCA/IA/AA combinations were chosen to compare with the conventional DMDHEU durable press finish or with the most effective nonformaldehyde durable press finish, BTCA. In general, the durable press finish of BTCA/IA/AA combinations did not provide as good whiteness indexes and durable press ratings for the finished fabric as those obtained from using the BTCA or the DMDHEU reactant.

A t-Test method is used for a pair-comparison between a selected BTCA/IA/AA finish formulation and with either BTCA alone or with DMDHEU. The finish formulations of 2.5%BTCA/8%IA/2.95%AA, with curing for 2 min. and with 1:1 mole ratio of monomers to catalyst (Test trial 1 finish formulation), and of 2%BTCA/9.6%IA/3.54%AA, with curing for 90 sec. and with 1:0.8 mole ratio of monomers to catalyst (Treatment #24 in Table 23) were chosen as the two BTCA/IA/AA combinations to be used in this discussion to compare the resultant

Table 23 Durable press finish formulations of the 32 treatments

Treatment No.	Reactant Concentrations (%o.w.b.)			Mole Ratio of Acid to Catalyst	Curing Time
	BTCA	IA	AA		
1.	2%	6.4%	1.77%	1:1	3 min.
2.	2%	6.4%	3.54%	1:1	3 min.
3.	2%	9.6%	1.77%	1:1	3 min.
4.	2%	9.6%	3.54%	1:1	3 min.
5.	2%	6.4%	1.77%	1:1	90 sec.
6.	2%	6.4%	3.54%	1:1	90 sec.
7.	2%	9.6%	1.77%	1:1	90 sec.
8.	2%	9.6%	3.54%	1:1	90 sec.
9.	3%	6.4%	1.77%	1:1	3 min.
10.	3%	6.4%	3.54%	1:1	3 min.
11.	3%	9.6%	1.77%	1:1	3 min.
12.	3%	9.6%	3.54%	1:1	3 min.
13.	3%	6.4%	1.77%	1:1	90 sec.
14.	3%	6.4%	3.54%	1:1	90 sec.
15.	3%	9.6%	1.77%	1:1	90 sec.
16.	3%	9.6%	3.54%	1:1	90 sec.
17.	2%	6.4%	1.77%	1:0.8	3 min.
18.	2%	6.4%	3.54%	1:0.8	3 min.
19.	2%	9.6%	1.77%	1:0.8	3 min.
20.	2%	9.6%	3.54%	1:0.8	3 min.
21.	2%	6.4%	1.77%	1:0.8	90 sec.
22	2%	6.4%	3.54%	1:0.8	90 sec.
23.	2%	9.6%	1.77%	1:0.8	90 sec.
24.	2%	9.6%	3.54%	1:0.8	90 sec.
25.	3%	6.4%	1.77%	1:0.8	3 min.
26.	3%	6.4%	3.54%	1:0.8	3 min.
27.	3%	9.6%	1.77%	1:0.8	3 min.
28.	3%	9.6%	3.54%	1:0.8	3 min.
29.	3%	6.4%	1.77%	1:0.8	90 sec.
30.	3%	6.4%	3.54%	1:0.8	90 sec.
31.	3%	9.6%	1.77%	1:0.8	90 sec.
32.	3%	9.6%	3.54%	1:0.8	90 sec.

Note. All the above durable press finish formulations also contained 1% polyethylene emulsion, 0.2% Triton X-100, and 1.5% o.w.m. potassium persulfate initiator, followed by predrying at 100 °C for 10 min. and curing at 180 °C for the times indicated above.

Table 24 Mean values of wrinkle recovery angle, breaking strength retention, tear strength retention, whiteness index, and durable press rating after one and five launderings for fabric specimens finished under 32 conditions involving BTCA/IA/AA combinations

Treatment Number	Wrinkle Recovery Angles (W+F)	Breaking Strength Retention W	Breaking Strength Retention F	Tear Strength Retention W	Tear Strength Retention F	Whiteness index	Durable press after Laundering 1x	Durable press after Laundering 5X
1.	266°	65	59	54	59	62	3.4	2.9
2.	252°	70	61	60	62	65	3.4	3.0
3.	276°	67	57	56	60	60	3.3	2.8
4.	276°	66	58	56	60	62	3.3	2.6
5.	254°	73	63	67	66	66	3.2	3.0
6.	257°	71	64	67	69	69	3.1	3.0
7.	268°	68	57	59	68	63	3.1	2.8
8.	267°	72	64	70	67	66	3.2	2.3
9.	273°	65	53	50	59	60	3.7	3.0
10.	280°	67	56	54	60	63	3.5	2.9
11.	285°	70	54	49	57	59	3.8	2.8
12.	284°	69	54	50	60	63	3.4	2.3
13.	270°	70	60	61	65	63	3.7	3.1
14.	264°	68	59	67	66	68	3.5	3.1
15.	279°	67	57	59	62	61	3.6	2.9
16.	272°	65	58	70	65	67	3.7	2.5
17.	269°	55	56	57	58	59	3.6	3.2
18.	267°	60	56	69	71	62	3.5	3.2
19.	273°	58	55	52	57	55	3.5	3.1
20.	278°	60	54	55	60	62	3.6	3.1
21.	259°	66	62	65	69	61	3.3	2.8
22.	256°	67	61	69	71	65	3.2	2.8
23.	267°	58	61	62	64	60	3.4	2.9
24.	265°	67	61	65	65	63	3.4	2.9
25.	273°	60	54	50	55	57	3.6	3.3
26.	275°	60	54	53	57	62	3.5	3.3
27.	280°	58	53	48	55	55	3.7	3.3
28.	282°	61	56	51	57	56	3.9	3.2
29.	266°	67	59	60	64	55	3.4	3.1
30.	263°	68	55	61	65	63	3.5	3.0
31.	278°	62	57	55	60	56	3.4	3.0
32.	272°	63	56	61	64	62	3.6	2.7

Note. The Processing conditions of each treatment are described in Table 23.

measured properties with those resulting from the 6.3% BTCA durable press finish. The above two BTCA/IA/AA finish formulations provided the strengths and wrinkle recovery properties of the finished fabric comparable with those of fabric finished with 6.3%BTCA (the most effective nonformaldehyde durable press finish). The raw data of each property resulted from the fabric finished with each of the above selected two BTCA/IA/AA finish formulations were compared with the raw data, using the t-Test, of the property resulted from the fabric finished with 6.3% BTCA. The results of the t-Test for a pair-comparison of the 2.5%BTCA/8%IA/2.95%AA (BTCA/IA/AA finish formulation in Trial 1) and the BTCA alone resulted that the properties of breaking strength (F (1,22) = 0.88, p = .3574), tear strength (F (1,12) = 0.64, p = .4402), wrinkle recovery angle (F (1,16) = 0.07, p = .7886), and durable press rating (F (1,32) = 0.53, p = .4705) of the fabric finished with either the BTCA/IA/AA finish formulation in Trial 1 or with the BTCA alone showed no significant differences from these two finish formulations, but the property of the whiteness index (F (1,10) = 319.20, p = .0001) was significant differences form these two treatments.

The results of the pair comparison of 2%BTCA/9.6%IA/3.54%AA (Finish formulation of BTCA/IA/AA in Treatment #24) and BTCA alone showed the wrinkle recovery angle (F (1,16) = 2.13, p = .1636) no significant differences from each other, but significant differences in other properties between these two finish formulations, the breaking strength (F (1,22) = 5.50, p = .0284), the tear strength (F (1,12) = 9.65, p = .0091), the whiteness index (F (1,10) = 197.22, p = .0001), and the durable press rating (F (1,32) = 22.00, p = .0001). The fabric finished with the BTCA/IA/AA combination might have yielded better results in the mechanical properties than did the BTCA-only when the concentrations among three reactants in the combinations were varied (as seen in the paired comparison of BTCA only and 2%BTCA/9.6%IA/3.54%AA).

According to the results of fabric specimens properties in Table 24, The whiteness index and the durable press rating properties of the fabric finished with BTCA/IA/AA combinations were overall not as good as those of the fabric finished with BTCA only. One of the above formulations, 2.5%BTCA/8%IA/2.95%AA, provided a good durable press rating compared to that of the BTCA reactant, but most of the BTCA/IA/AA combinations did not. The BTCA/IA/AA combinations need to be studied further to improve their ability in providing a better compromise between whiteness index and durable press rating. The research hypothesis 4 mentioned in the Chapter III addresses polymerization-crosslinking treatments using BTCA/IA/AA combinations with pad-dry-cure, and the BTCA reactant with pad-dry-cure will affect the mechanical and durable press properties and whiteness of treated fabrics differently. According to the results of the paired comparisons and results in Table 24, the research hypothesis 4 is partially accepted, because some of the analyzed BTCA/IA/AA combinations had the same effect on the wrinkle recovery angle and the mechanical properties (breaking and tear strength) as the BTCA reactant did. Most of the BTCA/IA/AA combinations affected the whiteness and the durable press rating differently from the BTCA reactant. The BTCA reactant, in

this study, provided better overall results on those two properties than the BTCA/IA/AA combinations did.

A few of the BTCA/IA/AA combinations provided a wrinkle recovery angles of the finished fabric that were close to that of fabric finished with DMDHEU (the conventional durable press finish). The 3%BTCA/9.6%IA/1.77%AA and 3%BTCA/9.6%IA/3.54%AA formulations, each with curing for 3 min. at 180°C and with either 1:1 or 1:0.8 mole ratio of monomers to catalyst, (Treatments # 11, 12, 27, and 28 in Table 23) provided wrinkle recovery angles of the finished fabric around 280° to 285°. These were comparable to the wrinkle recovery angle of the fabric finished with DMDHEU. The 3%BTCA/9.6%IA/1.77%AA, with 1:1 mole ratio of monomers to catalyst and curing time of 3 min. at 180°C (Treatment #11), was chosen as the BTCA/IA/AA combination, because it provided wrinkle recovery angle and breaking and tear strength of the finished fabric comparable with those obtained from the DMDHEU reactant. The raw data of the fabric finished with the above BTCA/IA/AA finish formulation were compared with those of the fabric finished with DMDHEU.

The finish formulation of BTCA/IA/AA combination in Treatment #11 provided a wrinkle recovery angle (F (1,16) = 3.03, p = .1010) and tear strength (F (1,12) = 0.03, p = .8590) properties of the finished fabric comparable to those of fabric treated with the DMDHEU. The breaking strength (F (1,22) = 11.96, p = .0022), whiteness index (F (1,10) = 374.73, p = .0001), and durable press rating (F (1,32) = 11.00, p = .0023) properties of the finished fabric with either the BTCA/IA/AA in Treatment #11 or with the DMDHEU showed significant differences from each other. According to the results in Table 24, the breaking strength of the fabric finished with the above BTCA/IA/AA combination was better than that using the DMDHEU reactant. The BTCA/IA/AA combinations did not provide good results in the whiteness index and the durable press rating areas to be comparable to the conventional DMDHEU reactant. The research hypothesis 5, mentioned in the Chapter III, addresses polymerization-crosslinking treatments using BTCA/IA/AA combinations with pad-dry-cure and the conventional DMDHEU treatment with pad-dry-cure, will affect the mechanical and durable press properties and whiteness of finished cotton fabrics differently. The results of the pair-comparison and in Table 24 could determine the partial acceptation of this hypothesis in that there were a few BTCA/IA/AA combinations that provided a wrinkle recovery angle and mechanical properties of the finished fabrics comparable to those treated with the DMDHEU reactant. Most of the BTCA/IA/AA combinations did not provide good results in whiteness and the durable press rating compared to those finished with the DMDHEU treatment.

CHAPTER VI
Summary and Conclusions

This chapter summarizes the research, the objectives of the research, and the interesting results of the study in the form of a manuscript that is required to be included in the dissertation.

Polymerization-Crosslinking Fabric Finishing, with Pad-Dry-Cure, Using Nonformaldehyde BTCA/IA/AA Combinations to Impart Durable Press Properties in Cotton Fabric

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ABSTRACT

This study examined the mechanical and durable press properties of cotton 3/1 twill-woven fabric finished with various concentrations in the BTCA/IA/AA reactants. The regression analysis was used to determine the relationship among each finishing variable, BTCA, IA, and AA concentrations, mole ratio of acid monomers to the sodium hypophosphite monohydrate catalyst, and curing time at 180 °C, and the finished fabric's property variables, breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press rating. Based on the results of the reduced R^2 regression equations and range dispersion of mean values of finished fabric properties. The results of the study indicated that some BTCA/IA/AA combinations applied to the cotton fabric provided good results in wrinkle recovery angle, breaking strength, and tear strength, comparable to those of the fabric finished with either the BTCA or DMDHEU reactant. The combinations of BTCA/IA/AA reactants did not provide as good whiteness and durable press rating properties as the BTCA or DMDHEU reactant.

Durable press finishing involves the use of chemical crosslinking agents that covalently crosslink with adjacent cellulose polymer chains within cotton fibers. The crosslinks result in the fabric's wrinkle resistance, but also in discoloration and impairment of fabric strength and of other mechanical properties. Dimethyloldihydroxyethyleneurea (DMDHEU) is the most widely used crosslinking agent because it provides good durable press properties at low cost and is less detrimental to fabric strength, discoloration, and other properties than other N-methylol agents; however, DMDHEU may release formaldehyde vapors during processing storage and consumer use. Largely because of concern about formaldehyde hazards to workers in the textile industry and also to consumers, formaldehyde-free crosslinking agents for producing durable press properties are of interest.

Several polycarboxylic-acid nonformaldehyde reactants have been studied to serve as durable press agents [1-8]. The main advantages of polycarboxylic acids are that they are formaldehyde-free, do not have a bad odor, and produce a very soft fabric hand. Loss of mechanical properties is a problem, however. The most effective of the polycarboxylic acid reactants is 1,2,3,4-butanetetracarboxylic acid (BTCA). BTCA, in the presence of sodium hypophosphite monohydrate as a catalyst, provides the same level of durable press performance and finish durability in laundering as does the conventional DMDHEU reactant. Yet, the high cost of BTCA

is an obstacle to mills' decisions to use BTCA as a replacement for the conventional durable press reactant. Mixed polycarboxylic acids in formaldehyde-free durable press finishing have been interesting research areas for decreasing the expense of using BTCA by reducing the required amount of BTCA in durable press finishing.

The present research was undertaken to examine the mechanical and durable press properties of cotton 3/1 twill woven fabrics treated by a polymerization-crosslinking process, with pad-dry-cure, using various concentrations of combinations of unsaturated carboxylic acids (itaconic acid, IA, and acrylic acid, AA) as polymer builders, and using BTCA as the crosslinker. This study determined the effects of several durable press finishing variables on key properties of the 3/1 twill finished fabric: breaking strength, tear strength, wrinkle recovery angle, whiteness index, and after one and five laundering cycles, durable press ratings. The durable press finishing variables to be examined were: (a) different concentrations of the unsaturated carboxylic acids (IA and AA), (b) different concentrations of BTCA, (c) different mole ratios of monomers to the sodium hypophosphite monohydrate catalyst, and (d) the different curing times. Regression analysis was used to estimate equations predicting breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press rating. The independent variables were the concentrations of BTCA, IA, and AA, the mole ratios of monomers to catalyst, and the curing times.

The regression analysis involved two steps. The first step was to run a full model with all the independent variables. The next step of the regression analysis was to simplify the full model by using the backward elimination procedure and maximum R^2 procedure. The R^2 is defined as the proportion of the variability in the dependent variable that can be predicted by the independent variables of a model [9]. The maximum R^2 procedure is defined as a method to find a high coefficient of determination that will be affected by the number of independent variables in a model. The reason for conducting these two procedures for current study was to find potentially simpler models that could predict the dependent variables nearly as effectively as the full model did.

The backward elimination procedure eliminates the least significant variables one by one from a full model until every variable left in the model is significant at the 0.10 level. The significant at the 0.10 level is set as the significant level instead of the 0.05 level in the backward elimination procedure because the higher level of significance allows the retention of more variables for models with interaction effects. The last model obtained from the backward elimination procedure for each dependent variable was run through the maximum R^2 procedure. This procedure indicated how R^2 was affected by the number of variables in the model. Based on the results of the maximum R^2 procedure, a model for each dependent variable was selected to have a reduced R^2 comparable to the R^2 of the full model. Predicted values from the two regression models for each of the dependent variables--the full model and the reduced R^2 model--were compared to the actual values of the property obtained from the actual measurements from the fabric finished with BTCA/IA/AA combinations. Only the regression equation with reduced R^2 was used for

interpreting the ability of each durable press finishing variable to affect the respective fabric property.

Experimental Procedure

Material: Scoured, bleached and mercerized 100% cotton 3/1 twill woven fabric was supplied by Cotton Incorporated. The fabric count was 104 x 48 per square 2.5 cm. (1 inch) and the fabric weight was 7.61 oz/yd^2, as measured by the researcher. The reactants used in the PCA finish formulations were laboratory-grade, at 99% purity, itaconic acid, acrylic acid, and 1,2,3,4-butanetetracarboxylic acid and were purchased from Aldrich Chemical Company, Inc. The acrylic acid contained 200 ppm hydroquinone monomethyl ether as an inhibitor to prevent polymerization. The amount of the initiator and catalyst used in the study were provided sufficiently to overcome the inhibitor, therefore, it was used in the study without removing the inhibitor. Dimethyloldihydroxyethyleneurea (DMDHEU) (Freerez^{TM} 900) was a commercial product in the form of a 44% solid solution, as supplied by Freedom Textile Chemicals Co. Laboratory-grade potassium persulfate initiator and sodium hypophosphite monohydrate catalyst, for finishing with PCA formulations, were purchased from Sigma Chemical Co. Laboratory-grade magnesium chloride hexahydrate catalyst, for finishing with DMDHEU, was purchased from Fisher Chemical. The nonionic wetting agent Triton X-100 was purchased from Rohm and Haas Co. The polyolefin emulsion softener (PAT-SOFT-PHD) was supplied by Yorkshire Pat-Chem.

Fabric Finishing: The cotton fabric was immersed in an aqueous finishing solution containing a mixture of IA and AA polymer builders, BTCA crosslinker, 1% polyolefin emulsion softener, 0.2% Triton X-100, sodium hypophosphite monohydrate catalyst, 1.5% o.w.m. potassium persulfate initiator, and water for 10 minutes. Each finishing formulation varied in the reactants' concentrations of the catalyst, and the initiator. The fabric was immersed and padded to effect a wet pick-up of 95-110% o.w.f., then mounted on a frame and predried at 100 °C for 10 minutes. The predried mounted fabric was removed from the oven, the temperature was raised to 180 °C, then the fabric was returned to the oven for curing at 180 °C for either 90 seconds or 3 minutes. After curing, the finished fabric was removed from the frame and rinsed in running hot tap water for 10 minutes, remounted on the frame and redried at 100 °C for 5 minutes.

For comparative purposes, the DMDHEU finish was applied as 12% DMDHEU, 1.5% magnesium chloride hexahydrate, 1% polyethylene emulsion, and 0.2% Triton X-100, followed by drying at 100 °C for 5 minutes and curing at 160 °C for 3 minutes. The BTCA-only finish was applied as 6.3% BTCA, 6.5% sodium hypophosphite monohydrate, 1% polyethylene emulsion, and 0.2% Triton X-100, followed by drying at 85 °C for 5 minutes. and curing at 180 °C for 90 seconds.

A total of 32 durable press finishing formulations of BTCA/IA/AA reactant combinations were used. The total of 32 was determined by calculating from two levels of each of the five durable press finishing variables in the study. Those five variables included: (a) two concentrations, 2% and 3% o.w.b., of BTCA, (b) two concentrations, 6.4% and 9.6% o.w.b., of IA, (c) two concentrations, 1.77% and 3.54% o.w.b., of AA, (d) two mole ratios, 1:1 and 1:0.8, of the acid monomers to the

sodium hypophosphite monohydrate catalyst, and (e) two curing times, 90 seconds. and 3 minutes. at 180 °C.

Methods for Measuring Fabric Properties: Breaking strength, tear strength, wrinkle recovery angle, durable press rating, and whiteness index were the five properties of the finished cotton fabric that were evaluated. Standard methods [10-12] were used to measure breaking strength (ASTM D 5035-95), tear strength (ASTM D 1424-83), and wrinkle recovery angle (AATCC 66-1990). An adaptation from the AATCC Test Method 124-1989 was used to rate durable press. In this study, the size of the specimens was reduced to 27.5 x 27.5 cm. because of the limitations of the equipment used in padding. The washing and evaluation procedures for AATCC Test Method 124-1989 were followed. This test method was used to determine how the crosslinking bonds formed in cellulose resisted bleaches, detergent, and distortion forces in the washing machine.

To perform the launderings before the durable press ratings, MAYTAG Model A806 was used as a washing machine and was made by the MAYTAG Company. The washing machine and dryer settings were followed for the normal or cotton/sturdy washing conditions mentioned in the AATCC 124-1989.

The whiteness index was measured on finished fabric specimens by AATCC Test Method 110-1989 using a Labscan Color Measuring Instrument made by Hunter Associates Laboratory, Inc. The x_n equal to 0.3152 and the y_n equal to 0.3346 were used in the whiteness index equation instead of the values given in the standard method because the instrument used in the study specified its own values for those two parameters. The breaking strength, tear strength, wrinkle recovery angle, and whiteness index were measured on the finished fabric without washing, whereas durable press ratings were made on the finished fabric after one and five launderings.

Results and Discussion

Table I summarizes the processing conditions for the applications of the 32 durable press finish formulations of BTCA/IA/AA reactant combinations. The mean values of the five properties of the specimens finished with each of the BTCA/IA/AA combinations were calculated and were used to find the dispersion range of the means for those five properties according to the durable press finishing variables. In Table II, the dispersion range, the fabric breaking strength, tear strength, wrinkle recovery angle, whiteness index, and durable press rating are reported.

By examining the dispersion ranges under each durable press finishing variable in Table II, it is apparent that those five variables can be divided into two groups. The first group variables contained BTCA concentration, IA concentration, and curing time. When the levels of those finishing variables in this group increased, they improved the wrinkle recovery angles and durable press ratings of the finished fabric, but reduced the breaking strength, tear strength, and whiteness index. The second group of finishing variables included the AA concentration and the mole ratio of monomers to catalyst. When the levels of those two variables increased, they improved the breaking strength, tear strength, and whiteness index of the finished fabric, but reduced the wrinkle recovery angles and durable press ratings of the finished fabric. The reduced R^2 regression equation for each fabric property of the

Table I. Durable press finish formulations of the 32 treatments.

Treatment No[a]	Reactant Concentrations (% o.w.b.)			Mole Ratio of Acid to Catalyst	Curing Time
	BTCA	IA	AA		
1.	2%	6.4%	1.77%	1:1	3 minutes
2.	2%	6.4%	3.54%	1:1	3 minutes
3.	2%	9.6%	1.77%	1:1	3 minutes
4.	2%	9.6%	3.54%	1:1	3 minutes
5.	2%	6.4%	1.77%	1:1	90 seconds
6.	2%	6.4%	3.54%	1:1	90 seconds
7.	2%	9.6%	1.77%	1:1	90 seconds
8.	2%	9.6%	3.54%	1:1	90 seconds
9.	3%	6.4%	1.77%	1:1	3 minutes
10.	3%	6.4%	3.54%	1:1	3 minutes
11.	3%	9.6%	1.77%	1:1	3 minutes
12.	3%	9.6%	3.54%	1:1	3 minutes
13.	3%	6.4%	1.77%	1:1	90 seconds
14.	3%	6.4%	3.54%	1:1	90 seconds
15.	3%	9.6%	1.77%	1:1	90 seconds
16.	3%	9.6%	3.54%	1:1	90 seconds
17.	2%	6.4%	1.77%	1:0.8	3 minutes
18.	2%	6.4%	3.54%	1:0.8	3 minutes
19.	2%	9.6%	1.77%	1:0.8	3 minutes
20.	2%	9.6%	3.54%	1:0.8	3 minutes
21.	2%	6.4%	1.77%	1:0.8	90 seconds
22	2%	6.4%	3.54%	1:0.8	90 seconds
23.	2%	9.6%	1.77%	1:0.8	90 seconds
24.	2%	9.6%	3.54%	1:0.8	90 seconds
25.	3%	6.4%	1.77%	1:0.8	3 minutes
26.	3%	6.4%	3.54%	1:0.8	3 minutes
27.	3%	9.6%	1.77%	1:0.8	3 minutes
28.	3%	9.6%	3.54%	1:0.8	3 minutes
29.	3%	6.4%	1.77%	1:0.8	90 seconds
30.	3%	6.4%	3.54%	1:0.8	90 seconds
31.	3%	9.6%	1.77%	1:0.8	90 seconds
32.	3%	9.6%	3.54%	1:0.8	90 seconds

[a]All the durable press finish formulations mentioned above also contained 1% polyethylene emulsion, 0.2% Triton X-100, and 1.5% o.w.m. potassium persulfate initiator, followed by predrying at 100 °C for 10 minutes.

Table II. The dispersion ranges of the fabric properties under each durable press finishing variable.

The first group of variables:

Fabric Property	Range Dispersion of Durable Press Finishing Variables: BTCA Concentration	IA Concentration	Curing Time
Breaking Strength (kg)			
Warp	-0.71 to +0.48	-0.82 to +0.51	-1.09 to +0.40
Weft	-0.48 to -0.01	-0.45 to +0.41	-0.54 to -0.01
Tear Strength (g)			
Warp	-312 to +16	-280 to +72	-288 to 0
Weft	-248 to +12	-200 to +32	-188 to 0
Wrinkle Recovery Angle	+4 to +28	+4 to +24	+2 to +16
Whiteness Index	-6 to 0	-6 to 0	-6 to -1
Durable Press Rating			
After the First Laundering	0 to +0.5	-0.1 to +0.4	0 to +0.3
After the Fifth Laundering	-0.3 to +0.3	-0.7 to +0.1	-0.5 to +0.2

The second group of variables:

Fabric Property	Range Dispersion of Durable Press Finishing Variables: AA Concentration	Mole Ratio of Monomers to Catalyst
Breaking Strength (kg)		
Warp	-0.19 to +0.93	+0.04 to +1.11
Weft	-0.24 to +0.47	-0.29 to +0.36
Tear Strength (g)		
Warp	-8 to +244	-168 to +124
Weft	-24 to +288	-160 to +88
Wrinkle Recovery Angle	-3 to +7	-4 to +2
Whiteness Index	+1 to +8	+1 to +8
Durable Press Rating		
After the First Laundering	-0.4 to +0.2	-0.5 to +0.3
After the Fifth Laundering	-0.5 to +0.1	-0.9 to +0.2

fabric finished with the 32 formulations of BTCA/IA/AA reactant combinations are summarized as follows. The independent variables (B, I, A, M, and C) in the equations represent the BTCA concentration (B), the IA concentration (I), the AA concentration (A), the mole ratio of monomers to catalyst (M), and the curing time (C).

The regression analysis for breaking strength produced two reduced R^2 equations because of the difference in the two directions of the measurements, warp or weft. The standard errors of the independent variables are shown in parentheses. The regression equation for the breaking strength (kg) for the warp direction (BSWA) is:

$$BSWA = 3.88 - 0.08(0.008)BC + 2.98(0.302)M \quad (1)$$

The regression equation for the breaking strength (kg) for the weft direction (BSWE) is :

$$BSWE = 4.78 - 0.08(0.008)BC \quad (2)$$

The reduce R^2 regression equation for the tear strength is the same for both the warp and weft directions. Thus, the fabric direction did not affect this property significantly. The data for tear strength for estimating the equation were in the units of percentage of the 3200-g total force used for testing. The regression equation for the tear strength (TS) is:

$$TS = 52.20 - 7.99(0.505)B - 0.38(0.072)I + 0.40(0.051)BA + 5.23(0.475)BM - 3.83(0.169)MC \quad (3)$$

The regression analysis for wrinkle recovery angle property produced three different reduced R^2 equations because of the differences for the fabric directions of the finished specimens and for the directions of the folded creases in the specimens used for measurements. The regression equation for the warp face to face wrinkle recovery angle (WAFF) is:

$$WAFF = 104.46 + 2.77(0.201)C + 0.56(0.026)BI \quad (4)$$

The regression equation for the warp back to back (WABB) and the weft face to face wrinkle recovery angle (WFFF) is:

$$WABB = WFFF = 125.33 + 2.77(0.201)C + 0.56(0.026)BI \quad (5)$$

The regression equation for the weft back to back wrinkle recovery angle (WFBB) is:

$$WFBB = 115.50 + 2.77(0.201)C + 0.56(0.026)BI \quad (6)$$

The reduced R^2 regression equation for the whiteness index (WI):

$$WI = 75.31 - 13.24(0.577)B - 0.58(0.082)I + 0.96(0.058)BA + 9.82(0.543)BM - 2.21(0.194)MC \quad (7)$$

The regression analysis for the durable press rating yielded two reduced R^2 equations, one for after one laundering and the other for after five launderings. The reduced R^2 regression equation for the durable press rating of the finished fabric after one laundering (DP1) is:

$$DP1 = 2.90 + 0.60(0.048)C + 0.16(0.024)BM - 0.56(0.051)MC \quad (8)$$

The reduced R^2 regression equation for the durable press rating of the finished fabric after five launderings (DP5) is:

$$DP5 = 2.90 - 0.05(0.005)I - 0.06(0.015)A + 0.60(0.048)C + 0.16(0.024)BM - 0.56(0.051)MC \quad (9)$$

From the simplified reduced R^2 regression equation for the breaking strength, it can be concluded that BTCA concentration and curing time were two main contributors to a decrease of breaking strength property of fabric finished with the BTCA/IA/AA combinations when the level of those above two variables increased. The mole ratio of acid monomers to catalyst contributed to an improvement of the finished fabrics. The IA and AA concentrations were excluded in the equations for breaking-strength property. That means the two variables did not significantly affect the breaking strength. The conclusion obtained from the simplified reduced R^2 equation of the breaking strength property was partially the same as those observed from dispersion ranges of the mean values.

From the simplified reduced R^2 regression equations for the tear strength and whiteness index, it can be concluded that the concentration of BTCA, the concentration of IA, and the curing time were the three main contributors to a decrease of those properties of the fabric finished with the BTCA/IA/AA combinations when the level of each variable in the finish formulation increased. The high level of AA concentration and mole ratio of monomers to catalyst variables contributed to an improvement of tear strength and whiteness of the finished fabrics. The conclusions obtained from the above simplified reduced R^2 regression equations for those fabric properties were the same as those observed from dispersion ranges of the mean values.

From the simplified reduced R^2 regression equation of wrinkle recovery angle, it can be concluded that BTCA and IA concentrations and curing time improved this property of the finished fabric, while AA concentration and mole ratio of monomers to catalyst did not affect the property significantly because they were not included in the equations of wrinkle recovery angle property. The conclusions obtained from the simplified reduced R^2 regression equations of the wrinkle recovery angle property were similar to some of those observed from dispersion ranges of the mean values.

For the durable press rating, the conclusion obtained from the regression analysis was different from that observed from dispersion ranges of the mean values. From the simplified reduced R^2 regression equation of durable press rating, BTCA and curing time improved the durable press rating of the finished fabric after both one and five launderings. The IA and AA concentrations decreased this property of the finished fabric after five launderings. These two variables were not included in the equation of durable press rating after one washing. They may affect the property but their effects were not significant. The mole ratio of monomers to catalyst could affect either the improvement or the deterioration the property depending on its interaction terms. The mole ratio of monomers to catalyst enhanced the ability of the BTCA concentration to improve the property, but it deteriorated the ability of the curing time to improve the property. From dispersion ranges of the mean values, the BTCA and IA concentrations and the curing time improved the property, but AA concentration and mole ratio of monomers to catalyst decreased the property.

From all above equations, each independent variable was taken the first derivatives to show a 1-unit change in each finishing variable affecting the fabric properties. Therefore, A summary of a 1-unit change in each finishing variable having the effects on the fabric properties is summarized below.

A 1-unit change in BTCA has these effects:

Breaking strength for the warp direction: -0.08C
Breaking strength for the weft direction: -0.08C
Tear strength for the both directions: -7.99 + 0.40A + 5.23M
Warp face to face wrinkle recovery angle: 0.56I
Warp back to back wrinkle recovery angle: 0.56I
Weft face to face wrinkle recovery angle: 0.56I
Weft back to back wrinkle recovery angle: 0.56I
Whiteness index: -13.24 + 0.96A + 9.82M
Durable press rating after one laundering: 0.16M
Durable press rating after five launderings: 0.16M

According to the above summary of the 1-unit change in BTCA, it can be concluded that increasing the 1-unit in BTCA concentration improved wrinkle recovery angle property of the finished fabric by 0.56 times IA concentration, and it improved durable press rating property by 0.16 times mole ratio of monomers to catalyst. The ability of BTCA to improve the wrinkle recovery angle and durable press rating properties depended on a variable of IA concentration and a variable of mole ratio of monomers to catalyst, respectively. The 1-unit change in BTCA decreased breaking strength of the finished fabric. A longer curing time enhanced the BTCA concentration to deteriorate the breaking strength more than the shorter curing time. Increasing 1-unit in BTCA tended to decrease tear strength and whiteness index properties. The ability of BTCA to deteriorate those two properties depended on the two variables of AA concentration and the mole ratio of monomers to catalyst.

A 1-unit change in IA has these effects:
Breaking strength for the warp direction: no effect
Breaking strength for the weft direction: no effect
Tear strength for the both directions: -0.38
Warp face to face wrinkle recovery angle: 0.56B
Warp back to back wrinkle recovery angle: 0.56B
Weft face to face wrinkle recovery angle: 0.56B
Weft back to back wrinkle recovery angle: 0.56B
Whiteness index: -0.58
Durable press rating after one laundering: no effect
Durable press rating after five launderings: -0.05

Increasing the 1-unit change in IA did not affect breaking strength and durable press rating after one laundering of the finished fabric, but it decreased the tear strength, whiteness index, and durable press rating after five launderings by -0.38, -0.58, and -0.05 unit, respectively. Increasing IA improved the wrinkle recovery angle and IA's ability to improve this property depended on BTCA concentration.

A 1-unit change in AA has these effects:
Breaking strength for the warp direction: no effect
Breaking strength for the weft direction: no effect
Tear strength for the both directions: 0.40B
Warp face to face wrinkle recovery angle: no effect
Warp back to back wrinkle recovery angle: no effect
Weft face to face wrinkle recovery angle: no effect

Weft back to back wrinkle recovery angle: no effect
Whiteness index: 0.96B
Durable press rating after one laundering: no effect
Durable press rating after five launderings: -0.06

Increasing the 1-unit change in AA concentration improved the tear strength and whiteness index by 0.40 and 0.96 times BTCA concentration, respectively. The BTCA enhanced the ability of AA concentration to improve the tear strength and whiteness index properties of the finished fabric, but the 1-unit change in AA decreased the durable press rating after five launderings by -0.06 unit. Increasing AA concentration did not affect the breaking strength, wrinkle recovery angle, and durable press rating after one laundering.

A 1-unit change in mole ratio of monomers to catalyst has these effects:

Breaking strength for the warp direction: 2.98
Breaking strength for the weft direction: no effect
Tear strength for the both directions: 5.23B - 3.83C
Warp face to face wrinkle recovery angle: no effect
Warp back to back wrinkle recovery angle: no effect
Weft face to face wrinkle recovery angle: no effect
Weft back to back wrinkle recovery angle: no effect
Whiteness index: 9.82B -2.21C
Durable press rating after one laundering: 0.16B - 0.56C
Durable press rating after five launderings: 0.16B - 0.56C

Increasing a 1-unit change in mole ratio of monomers to catalyst improved breaking strength for the warp direction by 2.98 unit, but it had no effect on breaking strength in the weft direction and wrinkle recovery angle properties. The increasing mole ratio of monomers to catalyst by 1-unit tended to affect the tear strength, whiteness index, and durable press rating properties either improving or decreasing depended on the finishing variables of BTCA and curing time. BTCA enhanced the ability of mole ratio of monomers to catalyst to improve those three properties, but curing time improved the ability of the mole ratio of monomers to catalyst to deteriorate those properties.

A 1-unit change in curing time has these effects:

Breaking strength for the warp direction: -0.08B
Breaking strength for the weft direction: -0.08B
Tear strength for the both directions: -3.84M
Warp face to face wrinkle recovery angle: 2.77
Warp back to back wrinkle recovery angle: 2.77
Weft face to face wrinkle recovery angle: 2.77
Weft back to back wrinkle recovery angle: 2.77
Whiteness index: -2.21M
Durable press rating after one laundering: -0.56M
Durable press rating after five launderings: -0.56M + 0.60

The 1-unit change in curing time improved wrinkle recovery angle property by 2.77 unit, but it tended to decrease the other properties. The ability of curing time to decrease the breaking strength property depended on the BTCA variable. The higher concentration of BTCA affected the breaking strength more than the lower concentration of BTCA. The variable of mole ratio of monomers to catalyst

enhanced the ability of curing time to deteriorate the tear strength, whiteness index, and durable press rating properties.

Based on the summaries of the 1-unit change in each finishing variable affecting the fabric properties, the mole ratio of acid monomers to catalyst variable positively affected the mechanical properties of the fabric finished with BTCA/IA/AA formulations, and it did not affect the wrinkle recovery angle property. It may be implied that if the fabric finished with BTCA/IA/AA formulation has the same level of the wrinkle recovery angle as that resulted from either BTCA alone or the DMDHEU, increasing the catalyst in a BTCA/IA/AA finish formulation could provide a better results of the mechanical properties of the finished fabric than those of the low catalyst concentration. In the case, the mechanical properties of fabric finished with BTCA/IA/AA formulation may be better than that of fabric finished with either BTCA alone or DMDHEU, when the wrinkle recovery angle of the fabrics finished with different durable press finishing agents was still remained comparable.

The AA concentration (A) affected in a positive trend of the tear strength and whiteness index properties of the finished fabric and did not affect breaking strength and wrinkle recovery angle properties. Therefore, increasing the AA in a BTCA/IA/AA formulation may improve a better tear strength and whiteness index properties of the finished fabric. The AA concentration variable negatively affected the durable press rating property of the finished fabric after the fifth laundering, so that increasing the AA concentration may improve the tear strength and whiteness index properties but it may decrease the durable press rating of the finished fabric at the same time.

It could be a possible way to compromise a good results in the wrinkle recovery angle, breaking strength, tear strength, and whiteness index of fabric finished with BTCA/IA/AA formulation to be comparable with those of a fabric finished with either BTCA only or the DMDHEU by adjusting the five finishing variables involved in the regression equations for the fabric properties.

Reasons that may support the above conclusions obtained from the range dispersions of the mean values of five fabric properties and the reduced R^2 regression equations are as follows:

(a) Increasing the concentrations of BTCA and IA, but not of AA, may increase the number of crosslinks formed in the cellulosic fibers. In general, for a given durable press finish reagent, the higher the concentrations of the reagents in the finish formulation, the larger the relative amounts of the reagents forming the covalent crosslinkages between the reagents and the cellulosic fibers. This phenomenon will improve the wrinkle resistance of the finished fabric. Fabrics that have the good crosslink networks in the fibers are expected to perform well both in wrinkle recovery angle and durable press rating. Fabrics having high wrinkle recovery angles should have high durable press ratings as well. Therefore, variables that improve the wrinkle recovery angle should enhance the durable press rating also. The crosslinking bonds formed in the cellulosic fibers should resist detergents, bleaches and distortion forces in washers and dryers, so that the bonds still can remain good crosslink networks and perform well in the durable press rating after laundering cycles.

(b) The formation of ester crosslinks in cotton fibers with a polycarboxylic acid requires heat and the presence catalyst [4-6]. The different mole ratios of acids to catalyst did not significantly affect the wrinkle recovery angle. In general, longer curing times and the higher curing temperatures effectively and maximally enhance the formation of ester crosslinks. Because the curing at 180°C was a constant temperature in this research, only the curing time could enhance the ester-crosslink mechanism. The longer the curing time used with the finish formulations, the greater number of crosslinks would be expected to form in the finished fabric, causing the fabric to be more wrinkle-resistant.

(c) Fabrics' loss of mechanical properties after durable-press finished can be ascribed to acid damage and the restriction of stress distribution within the fibers. The reagents used in the BTCA/IA/AA combinations are acids. Ester-crosslink networks impart resistance to the fabric wrinkling, but they increase the restriction of stress distribution within the cellulosic fibers. One could conclude that an increase of the concentration of BTCA, the concentration of IA, and the curing time may reduce the strength of the finished fabrics, in both the breaking and tear strengths. In addition, the high heat to which the cotton fabrics are exposed for a long time will scorch and reduce the strength of the fabrics. Therefore, the long curing time may cause a decrease in the mechanical properties and also whiteness of the finished fabrics.

(d) Yellowing of a durable-press to finished fabric may be caused by scorching of the fabric in the long exposure to a high temperature or may be caused by the number of double bonds in the IA/AA which has become attached to the finished fabric covalently, but which has not polymerized. According to results in a preliminary analysis in this research, adding acrylic acid in the finish formulation reduced the observed yellowing of the finished fabric. The acrylic acid reactant added to the mixture of the polymer builder system may enhance the polymerization of IA/AA mixture. Increasing the concentration of the acrylic acid may increase the rate of polymerization of the system of polymer builders; therefore, a polymer from the reaction may have a longer chain than that of the system having the low concentration of acrylic acid. The longer chain of the polymer may indicate that the monomers of IA and AA reactants were used up more than the shorter chain of the polymer, and it may be assumed that the finish formulation applied to the cotton fabrics has fewer double bonds in the system than that in the shorter chain of the polymer. The finished formulation having the smaller number of double bonds in the system applied to the cotton fabric may cause less yellowing or a higher whiteness on the finished fabrics.

(e) A longer chain of polymer may not only increase the whiteness but also may increase the strength of the finished fabrics because of less restriction of stress distribution inside the cotton fibers. Based on the results in this study, increasing the concentration of acrylic acid and the mole ratio of acids to catalyst were two main contributors to an improvement of the breaking strength, tear strength and whiteness properties of the finished fabrics. The greater amount of catalyst used in the finish formulations may improve the whiteness because it enhances the polymerization of the mixture of IA/AA as the AA reactant does enhance the polymerization of IA/AA mixture. The catalyst itself may have the ability to improve the whiteness of the

finished fabrics. These two main contributors decreased the wrinkle recovery angle and the durable press rating of the finished fabric perhaps because they may not enhance the ester-crosslink reaction, but they may increase the rate of polymerization of the mixture of IA/AA instead.

To assess the predictive ability of the two different regression equations--full model and reduced R^2-- estimated for each of the five fabric properties, the values of the properties predicted by those equations were compared with the actual mean values of each property obtained from the measurements in test trials. Table III summarizes the value of R^2 from the full model and the R^2 from the reduced model. Table IV summarizes the actual mean values and the predicted mean values from the full model and from the reduced R^2 model resulted from the four test trials' BTCA/IA/AA finishing formulations.

As seen in comparing the R^2 of those two models in Table III, the R^2 value of each of fabric properties from the reduced model is comparable with that from the full model. The differences of prediction in the mean values of fabric properties in Table IV are similar in those two models. The percent of actual mean values of two different models predicting the fabric properties are similar as well. The results of the predictions in Table IV could be concluded that the full model containing more variables did not predict the fabric properties better than the reduced R^2 model that contained fewer variables.

The mechanical and durable press properties of the fabric finished with BTCA/IA/AA combinations were compared with those finished with either the BTCA (the most effective nonformaldehyde durable press finish agent) or the DMDHEU (the most effective conventional durable press finish agent) reactant. Table V is the summary of the fabric properties of the fabric with no finish and when finished with BTCA or DMDHEU. Table VI is the summary of the mean values of fabric properties of the fabric finished with the BTCA/IA/AA reactant combinations. The finishing formulation of each treatment number is mentioned in Table I.

Most of the studied BTCA/IA/AA combinations provided breaking strength and tear strength comparable to those obtained from the BTCA reactant and to those obtained from the DMDHEU reactant. Some of the BTCA/IA/AA combinations provided good results in the wrinkle recovery angle compared to those obtained from the effective durable press finish agents.

The two finish formulations of 2%BTCA/9.6%IA/1.77%AA and 2%BTCA/9.6%IA/3.54%AA with curing for 90 seconds and with either 1:1 or 1:0.8 mole ratio of acids to catalyst (Treatments # 7, 8, 23, and 24), provided means wrinkle recovery angles around 265 to 268°, close to that obtained from the BTCA reactant. The 2%BTCA/6.4%IA/1.77%AA and the 2%BTCA/6.4%IA/3.54%AA (Treatments # 1, 17, and 18) provided wrinkle recovery angles close to that obtained from the BTCA reactant also, but those finish formulations needed to be cured for 3 minutes to get those angles. A few BTCA/IA/AA combinations provided good results in the wrinkle recovery angles and the mechanical properties compared with those provided by the DMDHEU reactant. The 3%BTCA/9.6%IA/1.77%AA and 3%BTCA/9.6%IA/3.54%AA, with curing for 3 minutes at 180 °C and with either 1:1 or 1:0.8 mole ratio of acids to catalyst (Treatments # 11, 12, 27, and 28), provided

Table III. The R^2 of full model and the R^2 of reduced model (maximum R^2) for the fabric properties.

	R^2	
Fabric Properties	Full Model	Reduced Model
Breaking strength	0.8744	0.8494
Tear strength	0.7963	0.7401
Wrinkle recovery angle	0.9320	0.9110
Whiteness index	0.8210	0.7978
Durable press rating	0.6634	0.5899

Table IV. Comparing actual mean values obtained from measurements with the predicted mean values by the full and reduced models from measurements in four test trials.

		Prediction from the Full Model			
	Actual	Predicted	Differences		% of actual
Fabric property	$\bar{X}$	$\bar{X}$	$\bar{X}$	S.D.	$\bar{X}$
Breaking strength (kg)					
Warp	6.18	6.23	0.05	0.0599	0.809
Weft	4.12	4.19	0.07	0.0294	1.699
Tear strength (g)					
Warp	33.03	36.31	3.28	0.5458	9.930
Weft	35.06	36.12	1.06	0.0049	3.023
Wrinkle recovery angle (°)					
	277	281	4.00	4.1477	1.444
Whiteness index	60	64.34	4.34	0.5152	7.233
Durable press rating					
After 1 laundering	3.56	3.68	0.12	0.0106	3.371
After 5 launderings	2.96	2.78	0.18	0.0639	6.081

		Prediction from the Reduced Model			
	Actual	Predicted	Differences		%of actual
Fabric property	$\bar{X}$	$\bar{X}$	$\bar{X}$	S.D.	$\bar{X}$
Breaking strength (kg)					
Warp	6.18	6.16	0.02	0.1404	0.324
Weft	4.12	4.30	0.08	0.0090	1.942
Tear strength (g)					
Warp	33.03	36.36	3.33	1.4705	10.082
Weft	35.06	36.36	1.03	0.0470	2.938
Wrinkle recovery angle (°)					
	277	278.29	1.29	0.7462	0.465
Whiteness index	60	64.30	4.30	0.4406	7.267
Durable press rating					
After 1 laundering	3.56	3.51	0.12	0.1103	3.371
After 5 launderings	2.96	2.77	0.19	0.1981	6.419

Table V. Mean value results of the fabric properties of the fabrics with no finish and when finished with BTCA or DMDHEU (retentions of breaking and tear strength are reported in parentheses).

	Fabric Finish		
Fabric Property	None	BTCA[a]	DMDHEU[b]
Breaking strength (kg) in warp	9.40	5.98 (64%)	5.76 (61%)
Breaking strength (kg) in weft	7.51	4.18 (56%)	3.99 (53%)
Tear strength (g) in warp	1944	1156 (59%)	952 (49%)
Tear strength (g) in weft	1852	1176 (63%)	1016 (55%)
Wrinkle recovery angle (°)	146°	268°	288°
Whiteness index	81	71	73
1st durable press rating	1.5	3.6	3.8
5th durable press rating	1.3	3.4	3.4

[a]The BTCA treatment is with 6.3% BTCA, 6.5% sodium hypophosphite monohydrate, 1% polyolefin emulsion, and 0.2% Triton X-100, followed by predrying at 85 °C for 5 minutes and curing at 180 °C for 90 seconds.

[b]The DMDHEU treatment is with 12% DMDHEU, 1.5% magnesium chloride hexahydrate, 1% polyolefin emulsion, and 0.2% Triton X-100, followed by predrying at 100 °C for 5 minutes and curing at 160 °C for 3 minutes.

Table VI. Mean values of wrinkle recovery angle, breaking strength retention, tear strength retention, whiteness index, and durable press rating after one and five launderings for fabric specimens finished under 32 conditions involving BTCA/IA/AA combinations. The processing conditions for each treatment are described in Table I.

Treatment Number	Wrinkle Recovery Angles (W+F)	Breaking Strength Retention W	Breaking Strength Retention F	Tear Strength Retention W	Tear Strength Retention F	Whiteness Index	Durable Press after Laundering 1x	Durable Press after Laundering 5x
1.	266°	65	59	54	59	62	3.4	2.9
2.	252°	70	61	60	62	65	3.4	3.0
3.	276°	67	57	56	60	60	3.3	2.8
4.	276°	66	58	56	60	62	3.3	2.6
5.	254°	73	63	67	66	66	3.2	3.0
6.	257°	71	64	67	69	69	3.1	3.0
7.	268°	68	57	59	68	63	3.1	2.8
8.	267°	72	64	70	67	66	3.2	2.3
9.	273°	65	53	50	59	60	3.7	3.0
10.	280°	67	56	54	60	63	3.5	2.9
11.	285°	70	54	49	57	59	3.8	2.8
12.	284°	69	54	50	60	63	3.4	2.3
13.	270°	70	60	61	65	63	3.7	3.1
14.	264°	68	59	67	66	68	3.5	3.1
15.	279°	67	57	59	62	61	3.6	2.9
16.	272°	65	58	70	65	67	3.7	2.5
17.	269°	55	56	57	58	59	3.6	3.2
18.	267°	60	56	69	71	62	3.5	3.2
19.	273°	58	55	52	57	55	3.5	3.1
20.	278°	60	54	55	60	62	3.6	3.1
21.	259°	66	62	65	69	61	3.3	2.8
22.	256°	67	61	69	71	65	3.2	2.8
23.	267°	58	61	62	64	60	3.4	2.9
24.	265°	67	61	65	65	63	3.4	2.9
25.	273°	60	54	50	55	57	3.6	3.3
26.	275°	60	54	53	57	62	3.5	3.3
27.	280°	58	53	48	55	55	3.7	3.3
28.	282°	61	56	51	57	56	3.9	3.2
29.	266°	67	59	60	64	55	3.4	3.1
30.	263°	68	55	61	65	63	3.5	3.0
31.	278°	62	57	55	60	56	3.4	3.0
32.	272°	63	56	61	64	62	3.6	2.7

wrinkle recovery angles of the finished fabrics around 280 to 285°, comparable to those of the fabrics finished with the DMDHEU reactant.

The drawbacks of the durable press finish of the BTCA/IA/AA combinations were that the fabric finished with these combinations did not yield good results in the whiteness index and durable press rating compared with those obtained from the BTCA or the DMDHEU durable press finish agent. The BTCA/IA/AA combinations need further study to improve properties of the whiteness and durable press rating of the finished fabric.

Summary and Conclusions

This research was conducted to test the combinations of BTCA/IA/AA reactants as a durable press finish to impart wrinkle resistance to cotton 3/1 twill woven fabric by the polymerization-crosslinking process with pad-dry-cure. The results of the study indicated that some BTCA/IA/AA combinations applied to the cotton fabric provided good results in wrinkle recovery angle, breaking strength and tear strength, comparable to those of the fabric finished with either the BTCA or DMDHEU reactant. However, the fabric finished with the BTCA/IA/AA combinations did not provide as good whiteness and durable press ratings as did the BTCA and DMDHEU reactants.

An adjustment of finishing variables, BTCA, IA, and AA concentrations, mole ratio of acid monomers to catalyst, and curing time, in a BTCA/IA/AA finish formulation, by using the information of the reduced R^2 regression equations of fabric properties, could provide the finished fabric properties differently. The wrinkle recovery angle of the fabric finished with BTCA/IA/AA formulation could be varied from a range of 265 to 268° to a range of 280 to 285° by varying the BTCA and IA concentrations, and curing time. The wrinkle recovery angles in the range of 265 to 268° are comparable to those of the fabric finished with the BTCA alone, while the wrinkle recovery angles in the range of 280 to 285° are comparable to those of the fabric finished with the DMDHEU. The mole ratio of acid monomers to catalyst and the AA concentration were excluded in the wrinkle recovery angle equations, therefore, adjusting either these two variables may not affect the wrinkle recovery angle property of the finished fabric, but they would affect the other fabric properties those involved with these two finishing variables. The variables of mole ratio of acid monomers to catalyst was involved in the breaking strength, tear strength, and whiteness index equations, and it affected positively those above fabric properties. An increasing the mole ratio of acid monomers to catalyst variable could provide a better results in mechanical and whiteness index properties of the finished fabric than those of the fabric finished at a lower concentration of the catalyst. The AA concentration as a finishing variable was included in the tear strength and whiteness index equations, and it affected in a trend of improving those two properties. An increasing the AA concentration could improve the tear strength and whiteness index properties.

The predictive ability of the two different regression equations was similar. The reduced R^2 model containing fewer variables predicted the fabric properties as effectively as the full model containing more variables did.

Further work related to this study should address the improvement of whiteness and durable press rating properties on fabric finished with combinations of BTCA/IA/AA reactants.

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CHAPTER VII
Implications and Suggestions for Future Research

This chapter indicates implications of this research and recommendations for further research related to this study.

Implications

This research was undertaken to analyze combinations of BTCA/IA/AA reactants with polymerization-crosslinking process and pad-dry-cure as durable press finishes to impart wrinkle resistance in cotton 3/1 twill woven fabric. It was concluded that some BTCA/IA/AA combinations applied to the cotton fabrics provided good results in wrinkle recovery angle and in breaking and tear strength, comparable to those of fabric finished with either the BTCA or DMDHEU reactant. The use of the mixture of IA/AA with BTCA in the finish formulations reduced the amount of BTCA reactant used; therefore, BTCA/IA/AA combinations may be alternatives to polycarboxylic acid derivatives that can serve for formaldehyde-free durable press agents at lower cost than a BTCA-based reactant. However, the fabrics finished with the BTCA/IA/AA combinations should not be used for any purpose that requires a high quality of whiteness or needs to be washed very often. The combinations of BTCA/IA/AA reactants did not provide as good whiteness and durable press rating properties as the BTCA and DMDHEU reactants did on the finished fabrics. Therefore, further work related to this study should address the improvement of whiteness and durable press rating properties on fabric finished with combinations of BTCA/IA/AA reactants.

The regression equations for measured properties of the finished cotton 3/1 twill woven fabric might be used to predict the values of properties of the fabric finished with other formulations of BTCA/IA/AA reactants. Some regression equations provided better predictions than the others depending on the R^2 of each equation. Higher R^2 equations better predict the properties than do lower R^2 equations. Because each analyzed independent variable was limited to only in two levels, the regression equations might predict more accurately the tested properties only within the range of the two analyzed levels of each of the independent variables.

Suggestions for Future Research

Based on the results of this study, the weak point of the durable press finish of the combinations of BTCA/IA/AA reactants was that the BTCA/IA/AA combinations did not provide results as good in the whiteness and durable press rating compared with those obtained from the BTCA or DMDHEU reactant. Not only did the BTCA/IA/AA combinations have the drawback in providing good whiteness, but the CA/IA/AA and BTCA/CA/IA combinations, which were studied only in the preliminary analysis, had the same problem also. The wrinkle recovery angle and breaking strength were the only two properties measured on the fabric finished with either the CA/IA/AA or the BTCA/CA/IA combinations in the preliminary analysis. Those two combinations provided good results in the wrinkle recovery angle and breaking strength, comparable with those obtained from the BTCA reactant. The durable press rating property was not measured in the preliminary analysis. These two combinations may or may not provide good results in the durable press rating for the

finished fabrics. If researchers can find a way to improve the drawbacks of the BTCA/IA/AA combinations, tests the CA/IA/AA and BTCA/CA/AA combinations should also be done. Therefore, the further study should be the search for means to improve the whiteness and durable press rating resulting with durable press finish agents of the BTCA/IA/AA, CA/IA/AA, or BTCA/CA/AA combinations.

A problem that may cause those two drawbacks of the fabric finished with the BTCA/IA/AA combinations may come from the double bonds of itaconic and acrylic acids left on the finished fabrics or the double bonds contained in the polymer. The double bonds are considered as a weakness that can cause degradation. The double bonds might react with bleaches or detergents and then depolymerize the bonds and form new shorter bonds, or form new oligomers in the cotton fibers after laundering. The new shorter covalent bonds or new oligomers formed in the finished fibers after laundering may not prevent the fabric from performing well in wrinkle resistance, compared with the effects of the bonds formed in the unlaundered finished fabrics. Therefore, further study should search for any method to improve the whiteness and durable press ratings of fabrics finished with combinations of polycarboxylic acid reactants. Suggestions for further work related to this study are as follows:

(a) Study the free-radical polymerization process of IA/AA or CA/AA by controlling the average molecular weight of a polymer. The average molecular weight of the polymer can indicate the length of the polymer chain. The variables that may affect the average molecular weight can be the concentrations of monomers, the types of initiators (potassium persulfate or others), the amount of initiator and catalyst used, and the temperature and time used in the free-radical polymerization process. This study may determine useful information about the relationship of the length of the polymer chain with the properties of the fabrics finished with various molecular weights of the polymers formed by different concentrations of combinations of IA/AA or CA/AA reactants. The results of the free-radical polymerization process of the combinations of IA/AA or CA/AA reactants may help to make a confirmed conclusion about whether the double bonds cause the problems of whiteness and durable press ratings of the fabric finished with the BTCA/IA/AA combinations or not.

(b) Study the effect of the boric acid treatment in the durable press finish agent of the BTCA/IA/AA, CA/IA/AA, or BTCA/CA/IA combinations. Welch et al. (1990) studied various treatments to remove the discoloration from print cloth treated with citric acid. Some of those treatments improved the whiteness index of the finished fabrics but deteriorated the durable press rating, but some of them did not. Boric acid was one of the treatments that improved the whiteness index of the finished fabrics treated with citric acid but did not affect the durable press rating on the finished fabrics.

(c) Study the effect of acids to sodium hypophosphite monohydrate ratios on the properties of the finished fabrics. Two different mole ratios of this effect were studied in this research, but the two different mole ratios (1:1 and 1:0.8) might not have been great enough to see an effect of this factor clearly. Andrews (1989, 1990) found that the fabric appearance, strength retention, and whiteness were improved when the acids to sodium hypophosphite monohydrate catalyst ratio was increased

for the citric acid system. Because this factor of acids to catalyst ratio improved those properties, it may improve the same properties on the fabrics finished with the BTCA/IA/AA combinations or other combinations of polycarboxylic acid reactants as well. The factor of acids to catalyst ratio should be expanded more than two levels in further study.

(d) Study the effect of just one finishing variable (e.g. curing time) at a time on the properties of the finished fabrics and use more than 2 levels of it (within the range and outside the range used in this research).

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Appendices A-V
Raw Data on the Measured Fabric Properties

Appendix A. Raw data on wrinkle recovery angles of fabric finished with the finish formulations and processing conditions for the experiments in Table 2

Experiment #	Average wrinkle recovery angles WF	WB	FF	FB	S.D.
1. Untreated	66°	71°	84°	70°	9.52
2. 12%DMDHEU	135°	152°	152°	136°	8.90
3. 6.3%BTCA	121°	142°	143°	131°	9.72
4. 2%BTCA/6%MA	124°	135°	138°	123°	7.14
5. 2%BTCA/6%IA	117°	139°	138°	122°	10.92
6. 2%BTCA/2.8%MA/3.2%IA	122°	139°	144°	125°	10.03
7. 2%CA/6%MA	116°	131°	133°	116°	8.78
8. 2%CA/6%IA	114°	131°	126°	118°	7.73
9. 2%CA/2.8%MA/3.2%IA	123°	127°	129°	118°	5.40
10. 2%SA/6%MA	93°	125°	124°	109°	11.76
11. 2%SA/6%IA	103°	123°	118°	112°	8.53
12. 2%SA/2.8%MA/3.2%IA	96°	116°	119°	101°	10.58

Note. WF, WB, FF, and FB stand for wrap face to face, warp back to back, weft face to face, and weft back to back, respectively.

Experiment #	Wrinkle recovery angle (°) Warp		Weft	
	Face to face	Back to back	Face to face	Back to back
1.	65,62,70	82,65,65	80,90,82	80,65,65
2.	135,135,135	150,155,152	152,152,152	132,138,138
3.	120,120,122	142,142,142	145,138,145	130,135,128
4.	122,125,125	132,138,136	140,138,135	125,122,122
5.	119,115,116	140,140,138	135,140,140	122,115,130
6.	120,125,120	138,140,138	145,142,145	130,125,120
7.	115,115,118	132,128,132	130,135,135	122,112,115
8.	112,112,118	130,132,132	130,130,118	120,118,118
9.	122,122,125	122,132,128	128,132,128	118,115,120
10.	100,98,100	122,132,120	125,128,120	106,110,110
11.	98,105,105	122,125,122	122,115,118	110,112,108
12.	92,95,102	120,112,115	115,120,122	102,102,98

Appendix B. Raw data on breaking strength of fabric finished with the finish formulations and processing conditions for the experiments in Table 2

Experiment #	Average breaking strength (kg) Warp	Weft	S.D.
1. Untreated	9.40	7.51	0.999
2. 12%DMDHEU	5.76	3.99	0.960
3. 6.3%BTCA	5.98	4.18	0.946
4. 2%BTCA/6%MA	6.21	4.16	1.107
5. 2%BTCA/6%IA	6.57	4.64	0.994
6. 2%BTCA/2.8%MA/3.2%IA	6.56	4.49	1.092
7. 2%CA/6%MA	6.31	4.55	0.906
8. 2%CA/6%IA	6.49	4.99	0.776
9. 2%CA/2.8%MA/3.2%IA	6.38	4.89	0.811
10. 2%SA/6%MA	6.77	4.98	0.953
11. 2%SA/6%IA	6.69	5.29	0.765
12. 2%SA/2.8%MA/3.2%IA	7.14	5.24	0.978

Experiment #	Breaking strength (kg) Warp	Weft
1.	9.2,9.3,9.7,8.8,10	7.5,7.6,7.8,7.5,7.5,7.5,7.4,7.3
2.	6.2,5.6,5.8,6.2,5.0	3.9,4.0,3.5,4.3,4.0,4.2,3.9,4.1
3.	5.6,5.8,6.3,5.9,6.3	4.0,4.6.4.4,4.0,4.1,4.0,4.1,4.2
4.	6.6,5.4,5.9,6.3,6.9	3.6,4.2,4.3,4.2,4.3,4.3,4.1,4.3
5.	6.8,6.5,6.7,6.6,6.2	4.7,4.7,4.8,4.8,4.7,4.4,4.4,4.7
6.	6.4,6.2,6.9,7.0,6.3	4.6,4.2,4.1,4.3,4.6,4.9,4.4,4.8
7.	6.4,6.5,6.4,6.0,6.2	4.7,4.6,4.4,4.3,4.5,4.5,4.8,4.6
8.	6.7,6.8,6.2,6.3,6.5	5.0,4.9,5.1,4.8,5.0,5.1,5.0,5.0
9.	6.1,6.3,6.6,6.2,6.7	5.6,5.0,5.1,4.8,4.8,4.6,4.8,4.5
10.	6.6,6.8,6.5,7.5,6.4	4.9,4.9,4.6,5.3,5.0,5.0,5.0,5.2
11.	6.5,6.3,7.2,6.4,7.0	5.2,5.5,5.2,5.5,5.0,5.5,5.5,4.9
12.	7.0,7.0,7.4,7.2,7.1	5.3,5.5,5.0,5.0,5.1,5.3,5.4,5.3

Appendix C. Raw data on wrinkle recovery angles of fabric finished with the finish formulations and processing conditions for the experiments in Table 3

Experiment #	Average of wrinkle recovery angle WF	WB	FF	FB	S.D.
1. 2%BTCA/6%MA	123°	139°	137°	132°	7.36
2. 2%BTCA/6%IA	112°	130°	127°	121°	7.68
3. 2%BTCA/2.8%MA/3.2%IA	118°	133°	130°	122°	6.92
4. 2%CA/6%MA	102°	122°	126°	103°	11.96
5. 2%CA/6%IA	98°	118°	122°	113°	10.42
6. 2%CA/2.8%MA/3.2%IA	104°	126°	127°	103°	12.15

Note. WF, WB, FF, and FB stand for wrap face to face, warp back to back, weft face to face, and weft back to back, respectively.

Experiment #	Wrinkle recovery angle (°)			
	Warp		Weft	
	Face to face	Back to back	Face to face	Back to back
1.	118,125,125	140,138,140	140,140,132	130,135,130
2.	115,112,110	130,135,125	130,125,125	120,126,118
3.	118,115,120	130,135,135	130,128,132	120,126,120
4.	102,98,106	128,122,115	122,128,128	98,106,105
5.	105,92,96	118,122,115	122,120,125	115,110,115
6.	100,106,106	125,128,125	125,125,130	100,108,102

Appendix D. The raw data on breaking strength of fabric finished with the finish formulations and processing conditions for the experiments in Table 3

Experiment #	Average breaking strength (kg) Warp	Weft	S.D.
1. 2%BTCA/6%MA	5.90	4.48	0.795
2. 2%BTCA/6%IA	6.36	4.89	0.785
3. 2%BTCA/2.8%MA/3.2%IA	6.24	4.76	0.808
4. 2%CA/6%MA	6.54	5.00	0.816
5. 2%CA/6%IA	7.04	4.96	1.119
6. 2%CA/2.8%MA/3.2%IA	6.42	4.94	0.807

Experiment #	Breaking strength (kg) Warp	Weft
1.	5.6,6.3,6.5,5.4,5.7	4.3,4.0,4.5,4.6,4.4,4.5,4.7,4.8
2.	6.2,5.8,6.6,6.8,6.3	4.8,5.2,4.7,4.9,5.0,5.0,4.8,4.7
3.	5.7,6.2,6.0,7.0,6.2	4.7,4.7,4.6,4.6,4.9,4.6,5.0,4.9
4.	6.5,7.1,6.6,6.1,6.4	4.9,5.0,4.8,5.2,5.0,5.0,5.2,4.9
5.	6.5,7.6,7.6,6.4,7.0	5.2,4.7,5.2,5.0,4.8,4.7,5.2,4.9
6.	5.9,6.3,6.5,6.4,7.0	5.1,5.2,4.8,5.2,4.6,5.0,5.0,4.6

Appendix E. The raw data on wrinkle recovery angles of fabric finished with the finish formulations and processing conditions for the experiments in Table 4

Experiment #	Average of wrinkle recovery angle WF	WB	FF	FB	S.D.
1. 2%BTCA/6%MA	110°	134°	135°	115°	12.29
2. 2%BTCA/6%IA	112°	132°	127°	114°	9.43
3. 2%BTCA/2.8%MA/3.2%IA	117°	134°	135°	116°	10.39
4. 2%BTCA/6%MA	111°	135°	132°	121°	10.61
5. 2%BTCA/6%IA	110°	127°	133°	112°	10.85
6. 2%BTCA/2.8%MA/3.2%IA	115°	133°	130°	116°	8.93

Note. WF, WB, FF, and FB stand for wrap face to face, warp back to back, weft face to face, and weft back to back, respectively.

Experiment #	Wrinkle recovery angle (°)			
	Warp		Weft	
	Face to face	Back to back	Face to face	Back to back
1.	105,110,116	135,130,138	130,132,142	116,115,113
2.	110,111,115	135,132,128	132,125,125	110,112,120
3.	112,116,122	130,135,138	135,134,136	110,122,115
4.	110,112,110	132,140,132	128,135,134	115,122,125
5.	105,110,115	130,120,130	135,132,132	115,112,108
6.	118,112,116	135,135,130	128,130,132	120,112,115

Appendix F. The raw data on breaking strength of fabric finished with the finish formulations and processing conditions for the experiments in Table 4

Experiment #	Average breaking strength (kg) Warp	Weft	S.D.
1. 2%BTCA/6%MA	5.79	4.57	0.655
2. 2%BTCA/6%IA	6.71	4.91	0.978
3. 2%BTCA/2.8%MA/3.2%IA	6.44	4.91	0.798
4. 2%BTCA/6%MA	6.31	4.63	0.873
5. 2%BTCA/6%IA	7.05	5.06	1.042
6. 2%BTCA/2.8%MA/3.2%IA	6.41	4.72	0.897

Experiment #	Breaking strength (kg) Warp	Weft
1.	5.7,5.6,6.0,5.9,5.7	4.6,5.0,4.4,4.7,4.5,4.8,4.4,4.2
2.	6.8,6.2,5.7,6.6,7.2	5.4,5.2,5.0,5.0,4.9,4.9,4.2,4.7
3.	6.5,6.4,6.56.5,6.3	4.5,5.0,4.7,5.1,5.0,4.7,5.1,5.2
4.	6.1,6.2,6.2,6.5,6.6	4.7,4.2,4.7,4.8,4.74.8,4.6,4.6
5.	6.8,7.1,7.1,7.6,6.6	5.0,5.1,5.1,5.2,5.0,5.0,5.3,4.8
6.	6.6,6.1,7.0,6.3,6.0	5.0,4.8,4.9,4.5,4.74.5,4.6,4.7

Appendix G. The raw data on wrinkle recovery angles of fabric finished with the finish formulations and processing conditions for the experiments in Table 5

Experiment #	Average of wrinkle recovery angle WF	WB	FF	FB	S.D.
1. 2%BTCA/2%CA/6%IA	122°	140°	140°	130°	8.55
2. 2%BTCA/2%CA/6%IA	122°	142°	142°	128°	9.50
3. 2%BTCA/2%CA/9%IA	125°	147°	148°	132°	10.42

Note. WF, WB, FF, and FB stand for wrap face to face, warp back to back, weft face to face, and weft back to back, respectively.

Experiment #	Wrinkle recovery angle (°)			
	Warp		Weft	
	Face to face	Back to back	Face to face	Back to back
1.	120,129,118	140,140,140	145,138,138	135,128,128
2.	122,120,125	140,145,140	142,138,145	122,128,135
3.	125,125,125	148,148,145	150,145,148	128,132,135

Appendix H. The raw data on breaking strength of fabric finished with the finish formulations and processing conditions for the experiments in Table 5

Experiment #	Average breaking strength (kg) Warp	Weft	S.D.
1. 2%BTCA/2%CA/6%IA	6.26	4.44	0.955
2. 2%BTCA/2%CA/6%IA	6.16	4.21	1.035
3. 2%BTCA/2%CA/9%IA	5.36	4.19	0.632

Experiment #	Breaking strength (kg) Warp	Weft
1.	6.2,6.2,5.7,6.7,6.5	4.4,4.5,4.5,4.3,4.4,4.2,4.4,4.8
2.	6.6,5.9,5.7,6.7,5.9	4.0,4.3,4.2,4.5,4.3,3.8,4.2,4.4
3.	5.3,5.2,5.3,5.7,5.3	3.9,3.9,4.0,4.3,4.2,4.5,4.2,4.5

Appendix I. The raw data on wrinkle recovery angles of fabric finished with the finish formulations and processing conditions for the experiments in Table 6

Experiment #	Average of wrinkle recovery angle WF	WB	FF	FB	S.D.
1. 2%BTCA/6%AA	93°	105°	115°	94°	9.46
2. 2%CA/6%AA	77°	106°	100°	85°	12.08
3. 2%BTCA/3.2%MA/1.77%AA	104°	125°	126°	113°	9.59
4. 2%BTCA/3.2%IA/1.77%AA	109°	122°	131°	112°	9.60
5. 2%BTCA/3.2%IA/1.77%AA	109°	127°	134°	115°	10.67
6. 2%BTCA/6.4%IA/1.77%AA	115°	136°	140°	121°	11.36
7. 2%CA/6.4%IA/1.77%AA	108°	130°	133°	112°	12.11
8. 4%CA/6.4%IA/1.77%AA	114°	138°	142°	122°	12.10
9. 3%BTCA/6.4%IA/1.77%AA	121°	144°	146°	124°	11.99

Note. WF, WB, FF, and FB stand for wrap face to face, warp back to back, weft face to face, and weft back to back, respectively.

Experiment #	Wrinkle recovery angle (°)			
	Warp		Weft	
	Face to face	Back to back	Face to face	Back to back
1.	92,95,92	105,108,102	115,115,115	95,92,96
2.	75,82,75	102,105,110	98,100,102	86,84,86
3.	100,108,105	125,125,125	125,125,128	115,115,110
4.	112,110,105	120,125,122	125,132,135	115,112,108
5.	102,110,115	125,130,125	135,135,132	115,115,115
6.	115,112,118	138,135,135	140,145,135	115,122,125
7.	105,108,110	130,135,125	132,130,138	115,112,108
8.	115,115,112	135,140,138	45,142,140	120,125,122
9.	118,120,125	142,145,145	145,145,148	125,125,122

Appendix J. The raw data on breaking strength of fabric finished with the finish formulations and processing conditions for the experiments in Table 6

Experiment #	Average breaking strength (kg) Warp	Weft	S.D.
1. 2%BTCA/6%AA	6.92	4.86	1.090
2. 2%CA/6%AA	7.26	5.58	0.906
3. 2%BTCA/3.2%MA/1.77%AA	6.02	4.81	0.697
4. 2%BTCA/3.2%IA/1.77%AA	6.44	4.77	0.921
5. 2%BTCA/3.2%IA/1.77%AA	5.94	4.49	0.786
6. 2%BTCA/6.4%IA/1.77%AA	5.90	4.49	0.762
7. 2%CA/6.4%IA/1.77%AA	6.34	4.49	0.986
8. 4%CA/6.4%IA/1.77%AA	5.95	4.25	0.966
9. 3%BTCA/6.4%IA/1.77%AA	6.04	4.14	0.998

Experiment #	Breaking strength (kg) Warp	Weft
1.	6.9,7.3,6.7,6.4,7.3	4.9,4.7,5.3,4.6,5.2,4.8,4.4,5.0
2.	7.0,7.7,6.8,7.4,7.4	5.4,5.8,6.0,5.5,5.2,5.8,5.7,5.2
3.	6.6,5.5,6.1,5.9,6.0	4.4,4.8,4.7,5.0,4.4,4.5,4.7,5.0
4.	6.5,6.3,6.3,5.9,7.2	4.4,5.1,5.1,5.0,4.4,4.5,5.0,4.6
5.	6.5,5.7,6.0,5.8,5.7	4.8,4.6,4.5,4.0,4.2,4.6,4.6,4.6
6.	6.0,6.4,5.6,5.8,5.7	4.0,4.6,4.9,4.5,4.5,4.4,4.5,4.5
7.	6.2,6.6,6.5,6.0,6.4	4.5,3.8,5.0,4.7,4.4,4.5,4.8,4.2
8.	7.0,5.8,6.0,5.4,5.5	4.5,4.1,4.2,3.6,4.5,4.5,4.3,4.1
9.	5.5,6.1,6.3,6.1,6.2	4.4,3.8,4.2,3.8,4.4,4.3,4.2,4.0

Appendix K. The raw data on wrinkle recovery angles of fabric finished with the 3%BTCA/6.4%IA/1.77%AA and processing conditions for the experiments in Table 7

3%BTCA/6.4%IA/1.77%AA	Average of wrinkle recovery angle WF	WB	FF	FB	S.D.
1. 1st fabric piece	118°	144°	145°	126°	12.86
2. 2nd fabric piece	117°	141°	142°	126°	11.55
3. 3rd fabric piece	119°	140°	143°	126°	10.66
4. 4th fabric piece	118°	140°	144°	124°	11.56
5. 5th fabric piece	118°	142°	147°	127°	12.51
6. 6th fabric piece	120°	142°	145°	125°	11.44

Note. WF, WB, FF, and FB stand for wrap face to face, warp back to back, weft face to face, and weft back to back, respectively.

Experiment #	Wrinkle recovery angle (°)			
	Warp		Weft	
	Face to face	Back to back	Face to face	Back to back
1.	120,118,115	140,148,145	148,142,145	125,120,132
2.	115,122,115	140,142,142	140,142,145	125,120,132
3.	122,120,115	138,140,142	142,142,145	132,125,122
4.	118,120,115	140,140,140	142,145,145	122,122,128
5.	120,115,118	140,142,145	145,148,148	130,125,125
6.	120,120,120	145,140,142	148,142,145	128,122,125

Appendix L. The raw data of breaking strength of fabric finished with the 3%BTCA/6.4%IA/1.77%AA and processing conditions for the experiments in Table 7

3%BTCA/6.4%IA/1.77%AA	Average breaking strength (kg) Warp	Weft	S.D.
1. 1st fabric piece	6.03	4.36	0.890
2. 2nd fabric piece	5.84	4.56	0.698
3. 3rd fabric piece	5.94	4.43	0.798
4. 4th fabric piece	6.18	4.52	0.882
5. 5th fabric piece	6.18	4.55	0.840
6. 6th fabric piece	6.16	4.56	0.823

Experiment #	Breaking strength (kg) Warp	Weft
1.	6.0,5.7,5.9,5.9,6.7	4.3,4.3,4.6,4.1,4.6,4.5,4.0,4.5
2.	6.4,5.8,5.9,5.2,5.9	4.5,4.7,4.6,4.7,4.7,4.4,4.6,4.3
3.	6.0,6.0,6.0,6.0,5.7	4.2,4.4,4.8,4.0,4.2,4.5,4.8,4.6
4.	5.5,6.5,6.4,6.5,6.0	4.6,4.7,4.7,4.4,4.6,4.5,4.3,4.4
5.	6.0,6.1,6.1,6.4,6.3	4.5,4.7,4.6,4.4,4.3,4.8,4.6,4.5
6.	6.0,6.2,6.1,6.20,6.3	4.5,4.3,4.5,4.5,4.6,4.5,4.9,4.7

Appendix M. Regression results of the last model given by the backward elimination procedure for the breaking strength (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	7.09	0.37	0.0001*
IA concentration	-0.18	0.04	0.0001*
curing time	-1.45	0.23	0.0001*
warp	-1.02	0.37	0.0055*
Interaction Terms			
BTCA*AA	-0.05	0.03	0.0534
BTCA*monomers/catalyst mole ratio	-0.42	0.12	0.0004*
BTCA*curing time	0.11	0.04	0.0063*
BTCA*warp	0.23	0.07	0.0011*
IA*AA	0.02	0.01	0.0080*
IA*curing time	0.04	0.01	0.0052*
monomers/catalyst mole ratio*curing time	0.69	0.14	0.0001*
monomers/catalyst mole ratio*warp	2.47	0.35	0.0001*

Note. The root mean square error of the full model = 0.3554.
R^2 = 0.8720
* $p < .05$

Appendix N. Regression results of the last model given by the backward elimination procedure for the tear strength (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	62.93	6.81	0.0001*
BTCA concentration	-7.20	1.92	0.0002*
IA concentration	-1.81	0.59	0.0025*
monomers/catalyst mole ratio	-15.15	7.51	0.0447*
warp	3.59	1.38	0.0101*
Interaction Terms			
BTCA*AA	0.23	0.06	0.0003*
BTCA*monomers/catalyst mole ratio	5.47	2.10	0.0097*
BTCA*warp	-1.16	0.42	0.0063*
IA*monomers/catalyst mole ratio	1.59	0.66	0.0163*
IA*warp	0.84	0.23	0.0004*
monomers/catalyst mole ratio*curing time	-3.02	0.22	0.0001*
curing time*warp	-1.48	0.28	0.0001*

Note. The root mean square error of the full model = 1.6790.
R^2 = 0.7901
* $p < .05$

Appendix O. Regression results of the last model given by the backward elimination procedure for the wrinkle recovery angle property (1 degree of freedom for every variable)

Variable	parameter Estimate	Standard Error	p-value
Intercept	149.12	3.39	0.0001*
monomers/catalyst mole ratio	-37.12	6.98	0.0001*
curing time	1.11	0.56	0.0475*
warp face to face	-27.62	3.16	0.0001*
weft back to back	-9.68	0.38	0.0001*
Interactions Terms			
BTCA*IA	-0.41	0.13	0.0022*
BTCA*monomers/catalyst mole ratio	8.61	1.22	0.0001*
IA*AA	0.15	0.08	0.0669
IA*monomers/catalyst mole ratio	2.47	0.43	0.0001*
IA*weft face to face	-0.35	0.17	0.0450*
AA*monomers/catalyst mole ratio	-2.73	0.83	0.0010*
AA*curing time	0.47	0.19	0.0158*
AA*warp face to face	-1.01	0.35	0.0043*
monomers/catalyst mole ratio*warp face to face	6.66	3.17	0.0364*
monomers/catalyst mole ratio*weft face to face	3.43	1.57	0.0300*
curing time*warp face to face	1.60	0.42	0.0002*

Note. The root means square error of the model = 2.6552.
$R^2 = 0.9305$
* $p < .05$

Appendix P. Regression results of the last model given by the backward elimination procedure for the whiteness index property (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	78.82	4.26	0.0001*
BTCA concentration	-16.14	2.03	0.0001*
IA concentration	-1.27	0.41	0.0022*
AA concentration	2.13	0.84	0.0123*
Interaction Terms			
BTCA*IA	0.28	0.16	0.0856
BTCA*AA	0.59	0.29	0.0413*
BTCA*monomers/catalyst mole ratio	10.04	0.92	0.0001*
BTCA*curing time	0.65	0.29	0.0274*
AA*curing time	-0.52	0.18	0.0048*
monomers/catalyst mole ratio*curing time	-2.48	0.89	0.0060*

Note. The root mean square error of the full model = 1.7710.
R^2 = 0.8139
* $p < .05$

Appendix Q. Regression results of the last model given by the backward elimination procedure for the durable press rating property (1 degree of freedom for every variable)

Variable	Parameter Estimates	Standard Error	p-value
Intercept	-1.62	0.63	0.0109*
IA concentration	0.38	0.06	0.0001*
AA concentration	0.39	0.12	0.0009*
monomers/catalyst mole ratio	4.04	0.70	0.0001*
curing time	0.85	0.14	0.0001*
fifth washings	1.55	0.24	0.0001*
Interaction Terms			
BTCA*monomers/catalyst mole ratio	0.37	0.07	0.0001*
BTCA*curing time	-0.0	0.02	0.0765
BTCA*fifth washings	-0.18	0.04	0.0001*
IA*AA	-0.01	0.01	0.0589
IA*monomers/catalyst mole ratio	-0.36	0.06	0.0001*
IA*fifth washings	-0.09	0.01	0.0001*
AA*monomers/catalyst mole ratio	-0.33	0.11	0.0036*
AA*fifth washings	-0.07	0.02	0.0020*
monomers/catalyst mole ratio*curing time	-0.72	0.13	0.0001*
monomers/catalyst mole ratio*fifth washings	-0.83	0.20	0.0001*

Note. The root mean square error of the full model = 0.2421.
$R^2 = 0.6517$
* $p < .05$

Appendix R. The raw data on breaking strength of fabric finished with each of 32 finish formulations of BTCA/IA/AA combination.

Experiment #	Breaking Strength (kg) Warp	Weft
1.	6.2,5.5,6.0,6.6,6.3	4.45,4.2,4.6,3.9,4.4,4.65,4.65,4.6
2.	6.6,6.6,6.3,6.7,6.65	4.8,4.7,4.1,4.9,4.6,4.4,4.8,4.6
3.	5.9,6.7,6.5,5.9,6.3	4.0,4.1,4.6,4.2,4.3,4.55,4.5,4.15
4.	6.5,6.1,6.9,6.4,5.3	4.6,4.65,4.2,4.3,4.34.1,4.1,4.4
5.	7.0,6.4,7.1,6.7,7.2	4.8,4.7,4.9,4.7,4.4,4.85,5.1,4.6
6.	6.9,7.05,6.05,6.6,7.0	4.95,4.7,5.1,4.7,5.0,5.0,4.65,4.6
7.	6.2,6.3,6.0,7.0,6.4	4.0,4.2,4.2,4.4,4.4,4.5,4.6,4.2
8.	7.0,6.6,6.4,7.1,6.85	5.2,3.9,5.15,4.6,4.7,4.95,4.7,5.0
9.	6.3,5.95,6.5,6.0,5.7	3.9,3.8,4.0,4.3,4.1,3.6,4.0,4.0
10.	5.4,6.0,6.9,6.8,6.2	4.3,4.3,4.6,4.0,4.0,3.8,4.6,4.2
11.	6.2,6.4,6.7,6.8,6.9	4.0,4.2,4.3,4.1,4.1,4.1,4.1,3.9
12.	6.2,6.45,6.8,6.8,6.1	4.3,4.0,4.2,4.4,3.5,4.2,4.2,4.0
13.	6.35,6.2,6.8,6.6,6.8	4.5,4.5,4.5,4.4,5.1,4.3,4.2,4.5
14.	6.85,6.6,6.2,5.9,6.4	4.6,4.0,3.8,4.4,4.9,4.3,4.6,4.7
15.	6.5,5.7,5.9,6.8,6.45	4.4,4.4,4.3,4.3,4.3,4.5,4.0,4.2
16.	5.5,5.6,6.8,6.9,5.6	4.8,4.7,4.4,4.35,4.2,4.0,4.3,4.2
17.	5.3,5.5,4.6,4.4,5.9	4.25,4.3,3.7,4.35,4.0,4.45,4.7,4.0
18.	5.8,5.2,5.7,6.1,5.4	4.1,3.8,4.3,4.3,4.5,4.5,4.0,4.5
19.	4.9,5.3,5.3,6.7,5.3	4.2,3.9,4.0,4.1,4.3,4.0,4.2,4.15
20.	5.8,5.6,5.7,5.4,5.55	4.0,4.5,3.9,4.3,4.15,3.9,4.1,3.9
21.	6.8,6.3,5.0,7.05,6.0	4.7,4.6,4.7,4.5,4.7,4.8,4.9,4.5
22.	5.6,6.7,6.3,6.5,6.4	4.6,4.6,4.7,4.7,4.5,4.6,4.8,4.4
23.	4.75,5.35,6.3,5.95,4.7	4.3,4.65,4.7,4.7,4.5,4.45,4.9,4.6
24.	6.2,6.4,6.6,7.3,5.2	4.9,4.85,4.3,4.75,4.8,3.9,4.75,4.6
25.	5.55,5.5,5.6,5.65,5.8	3.85,3.85,4.0,4.1,3.94.45,4.2,4.0
26.	5.65,6.0,5.2,5.85,5.5	4.2,4.0,3.8,3.8,4.2,4.15,4.2,4.1
27.	5.2,5.7,5.55,6.05,4.95	3.9,4.1,3.7,4.1,4.0,4.15,4.1,3.9
28.	6.75,5.2,5.45,5.4,6.1	4.3,4.2,4.45,3.95,4.0,3.9,4.4,4.3
29.	6.3,6.75,6.75,6.5,5.2	4.5,4.4,4.4,4.55,4.55,4.4,4.3,4.15
30.	6.1,6.3,6.2,5.8,7.35	3.8,4.35,4.5,4.0,4.4,4.0,4.1,4.2
31.	6.1,6.3,5.9,5.9,5.1	4.3,4.1,4.3,4.6,4.3,4.2,4.0,4.25
32.	6.4,5.6,6.2,5.9,5.5	4.3,4.55,4.3,4.4,4.4,4.0,3.9,4.0

Note. Processing conditions of each treatment are described under note in Table 23.

Appendix S. The raw data on tear strength of fabric finished with each of 32 finish formulations of BTCA/IA/AA combination.

Experiment #	Tear Strength (% of 3200 g) Warp	Weft
1.	35,30,33,32	34,32,34,36
2.	35,38,37,37	38,34,38,34
3.	33,33,35,35	35,34,34,36
4.	33,35,34,33	33,34,35,35
5.	38,37,43,46	35,39,40,40
6.	36,42,44,40	39,42,39,40
7.	35,36,35,38	40,39,40,39
8.	45,44,41,41	38,38,38,41
9.	31,29,31,30	32,35,35,34
10.	34,32,33,33	32,35,35,35.5
11.	29,28,29,32	33,33,33,33
12.	29,32,31,30	34,36,35,33.5
13.	40,39,35,35	38.8,40,37,36
14.	42,42,42,38	41,36,37,39
15.	38,35,35,36	36,37,35,36.5
16.	41.5,41.5,43,45	38,38,36.5,37.5
17.	34.5,32,36,35	34,33,33,35.5
18.	39,43.5,42,43.5	40,41,41,42
19.	32.5,33,30,31	33.5,33.5,32,34
20.	34,34,33,32	34.5,34.5,35,35
21.	37.5,39,41,41	40,39,41,39
22.	39,43.5,42,43.5	40,41,41,42
23.	38,35.5,38,39	38,36.5,36,36.5
24.	40,38,38,41.5	38,37,38.5,38
25.	30,30.5,31,31	33,33,31,30
26.	33,31.5,34.5,30	33.5,35,31.5,33
27.	28.5,27.5,30,30	33,31,31.5,32
28.	31.5,30,30,32	32.5,33,33,34
29.	35.5,36,36,38	36,37.5,37.5,38
30.	36,37,37.5,38	38.5,37.5,38,36
31.	33,34,33,33.5	35,35,35,34
32.	34.5,38.5,37.5,37.5	36,37,37,37.5

Note. Processing conditions of each treatment are described under note in Table 23.

Appendix T. The raw data on wrinkle recovery angle of fabric finished with each of 32 finish formulations of BTCA/IA/AA combination.

Experiment #.	Wrinkle recovery angle (°)			
	Warp		Weft	
	Face to Face	Back to Back	Face to Face	Back to Back
1.	122,122,120	135,138,138	140,145,142	132,135,130
2.	110,112,112	132,132,135	135,135,132	125,130,122
3.	128,128,125	145,145,145	145,145,145	135,135,132
4.	122,125,125	145,145,145	145,148,145	135,135,138
5.	115,115,112	135,135,135	135,135,132	125,125,128
6.	120,118,115	135,135,132	135,138,138	125,122,128
7.	118,120,122	142,142,142	142,142,142	132,135,131
8.	122,122,120	140,142,140	142,142,142	132,128,128
9.	125,125,120	142,145,145	145,145,145	135,135,132
10.	132,125,130	145,145,142	145,148,150	135,142,138
11.	132,130,135	152,148,150	150,152,148	135,138,138
12.	128,128,125	150,150,152	152,150,148	142,138,140
13.	125,122,120	140,145,142	145,140,142	132,135,135
14.	118,115,120	138,140,142	138,142,142	130,128,130
15.	122,130,128	145,142,148	142,145,145	135,135,138
16.	118,120,122	145,145,148	142,145,148	130,135,132
17.	125,125,122	142,140,138	140,140,140	132,135,135
18.	120,115,118	140,142,145	140,142,145	130,132,132
19.	122,125,125	145,148,145	142,142,142	135,135,132
20.	128,128,128	145,145,148	145,145,145	135,138,138
21.	118,115,115	140,140,138	140,140,142	122,125,120
22.	110,112,108	138,140,135	135,135,138	128,125,132
23.	115,118,120	142,142,140	145,140,142	135,132,132
24.	115,115,120	142,142,142	138,138,142	132,135,130
25.	128,125,125	145,142,140	145,145,142	130,135,135
26.	125,125,125	145,148,148	145,145,145	130,132,135
27.	125,130,128	145,145,148	145,145,148	140,142,140
28.	128,130,130	148,150,150	150,150,150	138,135,135
29.	118,118,115	138,145,140	142,145,140	130,132,130
30.	118,115,115	140,138,138	140,138,142	130,132,132
31.	125,128,128	145,148,148	145,145,142	135,138,138
32.	120,120,125	140,142,145	145,145,145	135,135,138

Note. Processing conditions of each treatment are described under note in Table 23.

Appendix U. The raw data on whiteness index of fabric finished with each of 32 finish formulations of BTCA/IA/AA combination

Experiment #	Whiteness index
1.	61.46,61.49,61.72,63.8,57.96,64.23
2.	66.62,63.53,65.13,64.52,64.59,65.16
3.	61.92,58.86,59.37,58.59,58.55,61.32
4.	61.19,63.35,63.65,64.16,60.49,62.34
5.	65.23,66.60,65.53,64.62,64.83,66.26
6.	68.51,68.73,68.65,68.94,69.65,69.30
7.	62.86,62.03,64.18,63.66,62.24,62.59
8.	66.84,66.35,66.76,64.92,66.36,66.55
9.	59.89,60.16,61.26,61.82,58.55,60.15
10.	62.86,62.03,64.18,63.66,62.24,62.59
11.	56.50,58.56,57.89,60.48,59.42,59.58
12.	61.93,64.46,62.17,62.26,65.09,62.17
13.	60.58,61.88,64.71,62.87,62.93,65.41
14.	69.25,67.87,67.41,68.74,69.27,68.50
15.	60.79,61.75,59.46,61.68,61.73,58.32
16.	67.86,67.40,66.84,66.39,67.16,66.89
17.	59.28,59.56,56.46,59.35,60.98,60.31
18.	61.72,63.10,60.81,62.60,60.62,62.55
19.	53.35,53.04,56.60,55.59,52.88,56.17
20.	61.39,60.60,61.06,61.58,64.03,62.16
21.	56.25,64.23,61.78,62.47,60.93,62.74
22.	65.36,64.24,66.93,65.22,66.00,65.01
23.	60.68,58.50,61.76,59.01,58.68,58.91
24.	62.28,64.95,63.09,62.80,65.10,62.24
25.	56.76,55.85,56.68,57.52,57.87,57.31
26.	60.89,62.26,61.54,60.85,62.88,61.65
27.	54.89,55.01,55.86,55.95,53.99,54.37
28.	54.52,57.29,56.13,57.43,56.30,56.41
29.	52.01,58.80,58.91,58.90,48.01,51.13
30.	63.41,61.49,62.75,62.04,63.40,63.77
31.	57.44,55.17,55.51,57.69,56.18,57.25
32.	63.15,63.82,59.60,64.04,63.22,60.00

Note. Processing conditions of each treatment are described under note in Table 23.

Appendix V. The raw data on durable press rating of fabric finished with each of 32 formulations of BTCA/IA/AA combination.

Experiment #	Durable press rating after One washing	Five washings
1.	3.5,3.5,3.5,3,3,3,3.5,3.5,4	3,3,3,3,3,3,3,2.5,3
2.	3.5,3.5,3,3.5,3.5,3.5,3.5,3,3.5	3,3,3,3,3,3,3,3,3
3.	3.5,3.5,3.5,3,3.5,3,3,3.5,3.5	3,3,3,3,3,2.5,3,2.5,2.5
4.	3.5,3,3.5,3,3.5,3.5,3,3.5,3.5	2.5,2,2.5,3,2,3,2,2.5,2.5
5.	3,3,3,3.5,3.5,3.5,3,3,3.5	3,3,3,3,3,3,3,3,3
6.	3,3.5,3,3,3,3,3.5,3,3	3,3,3,3,3,3,3,3,3
7.	3,3,3,3,3,3.5,3.5,3,3	3,3,3,2.5,2.5,3,2.5,3,3
8.	3.5,3,3.5,3,3,3,3.5,3,3.5	2.5,2.5,2,2,2.5,2.5,2.5,2,2.5
9.	4,4,3.5,3.5,3.5,4,3.5,3.5,3.5	3,3,3,3,3,3,3,3,3
10.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5	3,3,3,3,3,3,3,3,2.5
11.	4,3.5,4,4,3.5,3.5,4,3.5,4	3,3,3,3,3,3,2.5,3,2
12.	3.5,3.5,3.5,3.5,3,3,3.5,3.5,3.5	2.5,2,3,2,2.5,2,2,2,2.5
13.	3.5,3.5,4,3.5,3.5,3.5,4,3.5,4	3,3,3,3,3,3.5,3,3,3
14.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5	3,3,3,3,3,3,3.5,3,3
15.	3.5,3.5,4,3.5,3.5,3.5,3.5,3.5,3.5	2.5,3,3,3,3,3,3,3,2.5
16.	4,3.5,3.5,4,3.5,4,3.5,3.5,3.5	2.5,2.5,2.5,2.5,2.5,2,2.5,3,2.5
17.	3.5,3.5,3.5,3.5,3.5,4,3.5,3.5,3.5,3.5	3,3.5,3,3,3.5,3.5,3.5,3,3
18.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5	3,3.5,3.5,3,3,3,3,3.5,3
19.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5	3,3,3,3,3.5,3,3,3,3.5
20.	3.5,3.5,3.5,3.5,3.5,4,3.5,3.5,3.5	2.5,3.5,3,2.5,3.5,3,3,3.5,3.5
21.	3.5,3.5,3,3.5,3.5,3,3.5,3,3.5	3,2.5,2.5,3,3,3,3,3,2.5
22.	3.5,3,3,3.5,3.5,3,3,3,3	3,3,3,2.5,2.5,2.5,3,3,3
23.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3,3.5	2.5,3,3,3,3,3,3,3,3
24.	3.5,3,3.5,3.5,3.5,3.5,3.5,3.5,3.5	2.5,3,3,3,2.5,3,3,3,3
25.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5,4	3.5,3.5,3.5,3,3.5,3.5,3.5,3,3
26.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5	3.5,3.5,3,3.5,3,3,3.5,3.5,3
27.	4,3.5,4,3.5,3.5,3.5,4,4,3.5	3.5,3,3.5,3.5,3,3.5,3.5,3,3.5
28.	4,4,4,4,4,3.5,4,3.5,4	3,3,2.5,3.5,3.5,3,3.5,3.5,3
29.	3.5,3.5,3,3.5,3.5,3,3.5,3.5,3.5	3,3,3,3,3,3.5,3,3,3
30.	3.5,3.5,3,3.5,3.5,3.5,3.5,3.5,4	3.5,3,3,3,3,3,3,2.5,3
31.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,3.5,3	3,3,3,3,3,3,3,3,3
32.	3.5,3.5,3.5,3.5,3.5,3.5,3.5,4,4,3.5	3,3,3,3,2.5,3,2.5,2,2.5

Note. Processing conditions of each treatment are described under note in Table 23.

Vita

The author was born in Bangkok, Thailand. In 1984, she attended Chulalongkorn University in her hometown, where she received her Bachelor's Degree in the Department of Material Sciences, majoring in the Polymer and Textile Program. After graduation, she worked as a lab technician in the Futuretex Dyeing Company for 2 years. In 1990, she was selected by the office of the Civil Service Commission to take the Thai Government Scholarship to study aboard in the textile areas.

She came to the United States of America in Fall 1991 for her graduate work in Textile Chemistry at the University of Massachusetts at Dartmouth. She worked as a research assistant with the Gillette Company in the Department of Textile Science during her study there. In December 1993, she received her Master's of Science Degree in Textile Chemistry. Her thesis topic was "Elimination of the Drying Process before Dyeing using the Cold-Pad-Batch Dyeing Method."

In 1994, she entered graduate school in the Department of Clothing and Textiles at the Virginia Polytechnic Institute and State University (Virginia Tech) to pursue her doctoral degree in Textile Science. She completed her research work in Spring 1998. After graduation, she will go back to work as a lecturer in Chulalongkorn University, Department of Material Sciences, Bangkok, Thailand.

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